

FINAL**Long Term Monitoring Plan**

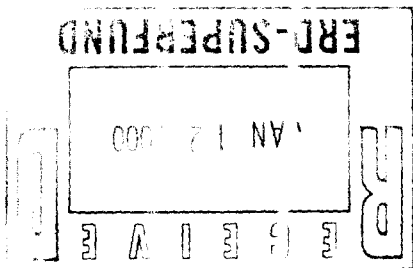
Allied Paper, Inc. / Portage Creek / Kalamazoo River
Superfund Site

Michigan Department of Environmental Quality
Environmental Response Division

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Contents



Contents

Section 1	Introduction	1-1
	1.1 Purpose of the Field Sampling Plan	1-1
	1.2 Area to be Investigated	1-2
Section 2	Work Plan Approach	2-1
	2.1 Project Scope and Objectives	2-1
Section 3	Sampling Tasks and Procedures	3-1
	3.1 Sampling Tasks	3-1
	3.2 Sampling Locations.	3-1
	3.3 Surface Water Sampling	3-2
	3.4 Resident Fish Sampling	3-5
	3.5 Sediment Sampling	3-6
	3.6 Caged Fish and Semipermeable Membrane Device Sampling	3-7
Section 4	Field Quality Assurance/Quality Control	4-1
	4.1 Sampling Documentation	4-1
	4.2 Sample Handling.	4-1
	4.3 Sampling Equipment Decontamination	4-3
	4.4 Quality Assurance Objectives	4-3
	4.5 Corrective Actions	4-6
	4.6 Quality Assurance Responsibilities	4-9
	4.7 Photograph Log	4-9
Section 5	Data Reduction, Validation, and Reporting	5-1
	5.0 Introduction	5-1
	5.1 Data Reduction	5-1
	5.2 Data Validation	5-3
	5.3 Laboratory Data Reporting	5-4
Section 6	Analytical Methods Summary	6-1

	6.1 Conventional Parameters	6-1
	6.2 Polychlorinated Biphenyls (PCBS)	6-1
Section 7	Project Schedule	7-1
Appendix A	Collection of Depth-Integrated Surface Water Samples for Organic Contaminant Analysis	
Appendix B	Resident Fish Collection by Boat-Mounted Electrofishing and Sample Processing for Organic Contaminant Analysis and GLEAS #31 Procedures	
Appendix C	SOPs for Collecting Bedded Sediment, Bedload Sediment, and Settling Sediment	
Appendix D	Bioaccumulation Studies Using Caged Fish	
Appendix E	Deployment of Semipermeable Membrane Devices	
Appendix F	Field Equipment Decontamination	

SECTION 1

Introduction

1.1 Purpose of the Long Term Monitoring Plan

The Michigan Department of Environmental Quality (MDEQ) requested that Camp Dresser & McKee (CDM) prepare a long term monitoring program that would accomplish the following:

- Develop a baseline data set for polychlorinated biphenyls (PCBs) in surface water, sediments, semi-permeable membrane devices, and fish tissue prior to remediation at selected locations in the Kalamazoo River and Portage Creek.
- Document and monitor levels of PCBs in abiotic and biotic media after the remediation activities have occurred.

Remediation of PCB contaminated sediments and flood plain soils has already been initiated on Portage Creek in Kalamazoo, Michigan and the Kalamazoo River near the Georgia-Pacific mill. The Long Term Monitoring Plan will address post-remediation sampling and monitoring at these sites, and develop a baseline data for other sites that will be remediated on the Kalamazoo River. Specific remediation activities will be site specific, and the goal of this Long Term Monitoring Plan is to develop a generic sampling strategy that would address different remediation scenarios for the Allied Paper, Inc./Portage Creek/Kalamazoo River (API/PC/KR) site.

The activities, methods, and schedules described in this Long Term Monitoring Plan are intended to address the first goal stated above; collection of baseline PCB contamination data from the API/PC/KR study area in 1999, prior to remediation. Individual remedial activities that will be undertaken in the future will have their own monitoring programs that will assess any short term benefits and/or impacts associated with the remediation. Thus, localized remediation monitoring - undertaken during and just after the remediation (i.e. performance standards)- is not covered in this FSP.

Sampling and monitoring will be conducted in conjunction with the MDEQ Surface Water Quality Division. The Surface Water Quality Division and their designated laboratory will be responsible for processing all fish, surface water and sediment samples for PCB analysis. Additionally, MDEQ will work closely with National Oceanic and Atmospheric Administration (NOAA) and the U.S. Fish and Wildlife Service (USFWS) during this long term monitoring effort to ensure their goals and objectives for this site are addressed.

Information gathered under the Long Term Monitoring Program for the API/PC/KR site, will be formatted so that the data and supporting information can be incorporated into a MDEQ Web-based site, that can be accessed by the public and interested parties. CDM will prepare the Web-site when directed by MDEQ.

1.2 Proposed Sampling Area

The area to be investigated in this first year extends from Ceresco Reservoir (at the upstream end) downstream to just above Saugatuck (**Figure 1.1**). Section 3 describes the individual stations to be sampled, the rationales for their choice, the sample types to be collected at each station, and the collection frequencies.

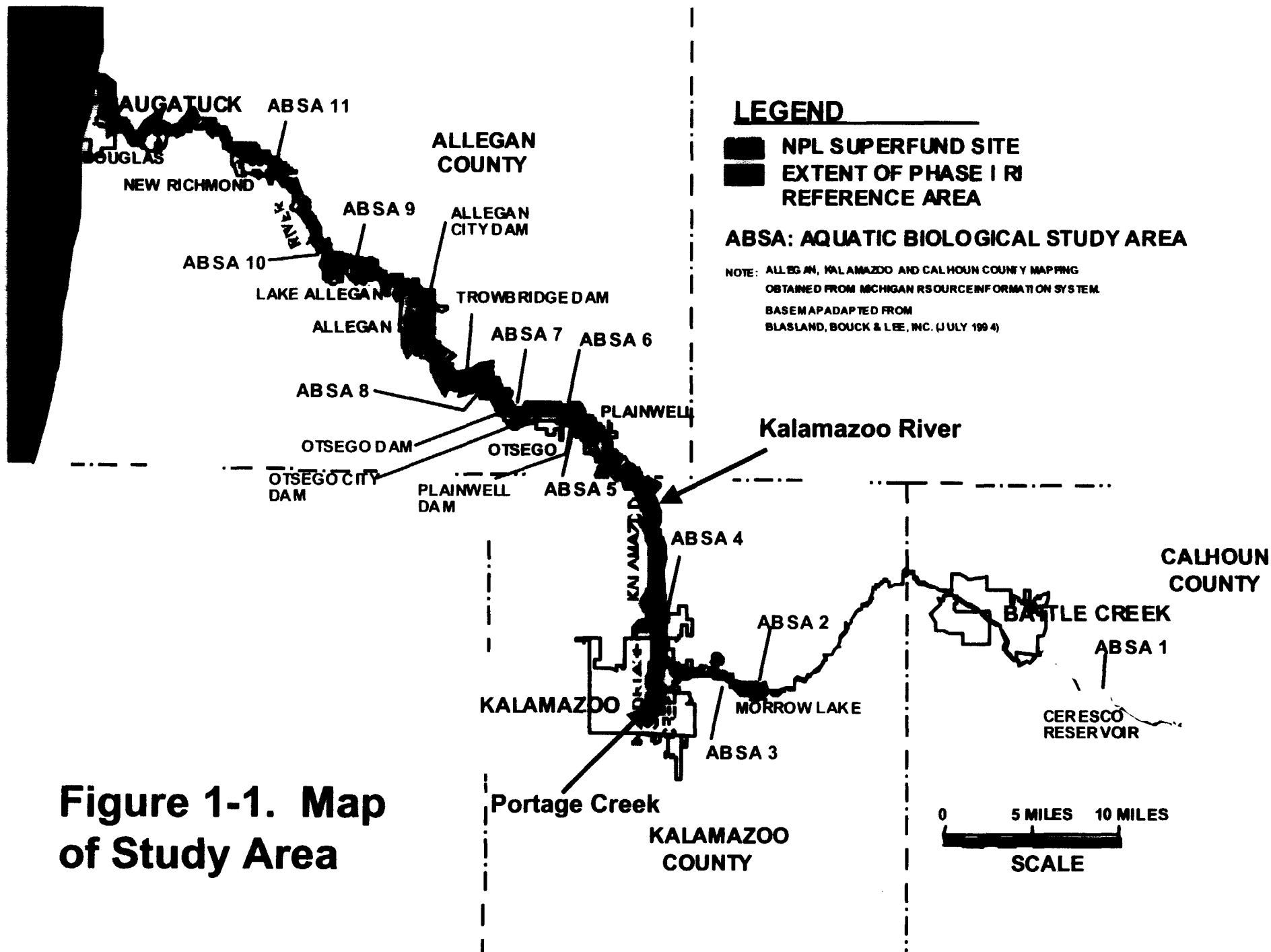


Figure 1-1. Map of Study Area

SECTION 2

Work Plan Approach

2.1 Project Scope and Objectives

The general objective of the activities and schedules of this Field Sampling Plan is to establish the nature and extent of polychlorinated biphenyl (PCB) contamination in the API/PC/KR study area, prior to environmental remediation activities. Numerous studies over the years have documented high PCB concentrations in water, sediment, soil, and fish from the study area. However, spatial and temporal coverage of the Site is incomplete, and the quality of the analytical data in some previous studies is inadequate. These problems combine to limit the usefulness of existing PCB data sets.

The objectives of this investigation include the following:

- Establish baseline (pre-remedial action) PCB concentrations in surface water, fish, and sediments collected from within the study area.
- Evaluate PCB concentrations upstream of the study area.
- Investigate the seasonal variation in water column PCB concentrations (summer vs. winter; dry weather vs. wet weather), to establish the importance of temporally variable contaminant bioavailability due to bioturbation and other biological activity, sediment resuspension during rain events, etc.

The activities implemented to achieve these objectives include:

- Sampling surface water, resident fish, sediments of several types, and caged fish and semipermeable membrane devices (SPMDs) from selected locations in the study area.
- Analyzing these samples for individual PCB congeners. Data for total PCBs are then obtained from the sum of the individual congener concentrations. Note that analysis of PCBs by congener not only allows for lower detection limits to be obtained (relative to Aroclor analyses) but also generally allows for more accurate analyses (including total PCB values) and it provides a better basis for understanding PCB fate and transport in the environment.

- Examining the PCB congener data using chemical "fingerprinting" statistics, to investigate (1) possible contaminant sources, and (2) possible congener-specific toxicity.
- Using statistical techniques to permit comparison of the congener-specific data to the PCB Aroclor data generated in previous studies.
- Sampling yearling fish (smallmouth bass), and caged channel catfish and semipermeable membrane devices, to assess year-to-year changes in water quality and monitor the effectiveness of remedial activities.

More detailed descriptions of these activities follow in Section 3.

SECTION 3

Sampling Tasks and Procedures

3.1 Sampling Tasks in Year 1 (1999)

During the first year of the Long Term Monitoring Program (1999), the following sampling activities will be conducted:

- Surface water sampling, to document the distribution of PCBs in water during dry and wet weather conditions.
- Sediment sampling, to assess the distribution of PCB-contaminated sediments prior to remediation activities. Bedded sediment samples will be collected from several impoundments in the study area. Bedload sediment will be collected from additional locations, to qualitatively assess the importance of near-bottom transport of PCB-contaminated sediments under wet weather conditions. Settling sediment will be sampled using sediment traps at all impoundments (Ceresco, Morrow Pond, Plainwell Dam, Otsego City Dam, Otsego Dam, Trowbridge, and Lake Allegan).
- Resident fish sampling and caged channel catfish and semipermeable membrane device (SPMD) deployments, to assess bioavailable PCB concentrations. Resident adult carp and smallmouth bass will be collected to augment the data collected during previous trend monitoring activities on the Kalamazoo River. Contaminant data for resident adult fish provide an assessment of both ecological and human risk as well as an account of historic PCB contamination. In addition, yearling smallmouth bass and caged channel catfish and SPMDs will be collected from the same locations as the adult fish, to assess current bioavailable PCB concentrations.

The locations at which these sampling activities will take place are described in Section 3.2, and details of their execution are provided in Sections 3.3 to 3.6.

3.2 Sampling Locations

Samples of one type or another will be collected at 23 stations in 1999. These stations were selected with the following general objectives in mind:

- To bracket potential PCB sources *areas (e.g. Plainwell Dam impoundment)*
- To resample locations sampled in previous studies, for trend analysis

- To sample at important recreational areas
- To sample areas most likely to have PCB-contaminated sediment deposits
- To sample areas presumably reflective of "background" conditions

Surface water samples will be collected at 18 locations under dry weather conditions, and at a subset of 10 of these locations under wet weather conditions (**Table 3.1**). The wet weather locations were chosen to isolate potential and/or known sources of PCB loadings to the river.

Bedded sediment, resident fish, caged SPMDs, and caged fish will be collected at a subset of dry weather surface water stations (**Table 3.2**) in the late summer/early fall of 1999. Most of these locations are impoundments, which are the areas that have accumulated high amounts of PCBs, and it is anticipated that these areas will be remediated. Bedload sediment samples will be collected at 5 locations, and settling sediment will be collected at the six impoundments. Both bedload and settling sediments will be collected under wet weather conditions.

Data collected during 1999 will be evaluated before formulating monitoring plans for 2000 and 2001. Particular attention will be paid to:

- Spatial and temporal heterogeneity of PCB concentrations in water
- Correlations of PCB concentrations in different media, especially between resident fish, caged fish, and semipermeable membrane devices.

3.3 Surface Water Sampling

Water samples will be collected at 18 locations under dry weather (baseflow) conditions and at 10 locations under wet weather conditions (**Table 3.1**). Three dry weather surveys and 2 wet weather surveys will be conducted in 1999. One-liter samples will be collected at each location by submerging the sample bottle directly into the water column, as per the Standard Operating Procedure (SOP) in Appendix A. A cross-channel transect will be established at each sampling location. Three individual samples will be collected along the transect, at mid-channel and at two locations close to each bank. These near-shore locations will be nominally located at 10 percent and 90 percent of the transect width, unless plumes of suspended sediment are visible closer to bank, in which case the near-shore locations will be positioned in the middle of the plume. Samples will be depth-integrated by use of a "filler cap", if necessary, as described in the SOP (**Appendix A**).

At locations where other sample types will also be collected (e.g., sediments or fish), the water samples will be collected first, to minimize the possibility of contaminating the water sample. In the wet weather surveys, water samples will be collected at mid-channel from the bridges.

Water samples will be analyzed for PCB congeners, as well as water temperature and total suspended solids. In addition, river stage readings will be made at four permanent staff gages - those in Battle Creek, Comstock, and Portage Creek - during each water sampling survey. These stage readings will be used to estimate river flow from existing stage-flow relationships.

**Table 3.1 Sampling Matrix for Year 1 (1999):
Surface Water, and Bedload & Settling Sediment (Number of Samples)**

Location		Surface Water (DW)	Surface Water (WW)	Bedload Sediment	Settling Sediment
1.	Ceresco Reservoir	9			
2.	Morrow Lake	9	2	2	3
3.	Kalamazoo River, near Comstock	9			
4.	Portage Creek, at Kilgore Road	9			
5.	Portage Creek, at Alcott Street	9	2	2	
6.	Portage Creek, at Kalamazoo Avenue	9			
7.	Kalamazoo River, at King Hwy.	9	2		
8.	Kalamazoo River, at D Ave.	9	2		
9.	Kalamazoo River, at U.S. 131	9	2	2	3 Plainwell Impoundment
10.	Kalamazoo River, Downstream of Plainwell Dam	9			
11.	Kalamazoo River, at Farmer Street (Otsego)	9	2		3 Otsego City Impoundment

**Table 3.1 Sampling Matrix for Year 1 (1999):
Surface Water, and Bedload & Settling Sediment (Number of Samples)**

	Location	Surface Water (DW)	Surface Water (WW)	Bedload Sediment	Settling Sediment
12.	Kalamazoo River, Downstream of Otsego Dam	9			3 Trowbridge Impoundment
13.	Kalamazoo River, at 26 th St.	9			
14	Kalamazoo River, at Highway 118 (Allegan)	9	2	2	3 Allegan City Impoundment
15.	Lake Allegan	9			3
16.	Kalamazoo River, at 124 th Ave.	9	2		
17.	Kalamazoo River, at 58 th Street (near New Richmond)	9	2	2	
18.	Kalamazoo River, at Blue Star Hwy (Douglas)	9	2		
Total		162	20	10	18

DW = under dry weather conditions; WW= under wet weather conditions

**Table 3.2 Sampling Matrix for Year 1 (1999)
Resident and Caged Fish, SPMDs, and Bedded Sediment
(Number of Samples)**

	Location	Resident Fish *	Caged Fish & SPMDs	Bedded Sediment/ Water
1.	Ceresco Reservoir	11A+5Y	3 each	1 composite sample each
2.	Morrow Lake	11A+5Y	3 each	1 composite sample each

3.	Portage Creek, Upstream of Alcott St.	11A+5Y (yearling SMB may not be available, other species may be substituted)	3 each	1 composite sample each
4.	Kalamazoo River, Upstream of Plainwell Dam	11A+5Y	3 each	1 composite sample each
5.	Kalamazoo River, Upstream of Otsego City Dam	11A+5Y	3 each	1 composite sample each
6.	Kalamazoo River, Upstream of Otsego Dam	11A+5Y	3 each	1 composite sample each
7.	Kalamazoo River, Upstream of Trowbridge Dam	11A+5Y	3 each	1 composite sample each
8.	Kalamazoo River, Upstream of City of Allegan Dam	11A+5Y	3 each	1 composite sample each
9.	Lake Allegan	11A+5Y	3 each	1 composite sample each
10.	Kalamazoo River, at 58 th Street (near New Richmond)	11A+5Y		1 composite sample each
11	Kalamazoo River near Kalamazoo Lake	11A+5Y	3 each	1 composite sample each
<i>Total</i>		12 1A + 55Y 2A2	30 of each	11 composite sample

* A = Adult Carp and Smallmouth Bass (fillet and remaining carcass processed individually); Y - Yearling Smallmouth Bass (composite = 3-5 fish; whole body analyzed)

3.4 Resident Fish Sampling

Adult resident fish will be collected from 11 locations and yearling fish will be collected from 11 locations (**Table 3.2**). Resident adult carp and smallmouth bass, and yearling smallmouth bass, will be collected by electroshocking from a small boat, operated as per the SOP included in **Appendix B**. Eleven adult fish of

(Carp) skin-on smallmouth bass

each species will be collected and individually processed into skin-off fillet and fillet-less carcass samples. Multiple composite samples consisting of three-five whole body yearling smallmouth will be collected from each station in **Table 3.2**.

Fish samples will be collected once during 1999, in late summer/early fall near the end of the caged SPMD exposure period. Adult fish will be analyzed for total PCBs and lipid content, while yearling smallmouth bass will be analyzed for PCB congeners and tissue lipid content. The purpose of analyzing yearling smallmouth bass for PCB congeners is to further identify additional sources of PCB loadings to the river after remediation has occurred. All fish will be processed by the MDEQ Surface Water Quality Division, and fillet tissues will be analyzed for total PCBs and lipids by their designated laboratory. Whole-body yearling smallmouth bass composites will be analyzed for PCB congeners and lipids by the laboratory contracted to MDEQ.

3.5 Sediment Sampling

Three types of sediment samples will be collected; bedded sediments, bedload sediment, and settling sediment; at the stations listed in **Tables 3.1 and 3.2**. SOPs describing each sediment sampling technique are included in **Appendix C**.

Bedded sediment - sediment deposited on the stream bed - will be collected using 2-meter lengths of cellulose acetate butyrate (CAB) plastic core tubes. A battery-powered, hand-held vibrocore unit will be used to drive the core tubes into the sediment until refusal (i.e., until the core tube no longer penetrates the sediment). Core tubes will be cut open and the upper 6" collected. This is believed to be the biologically active sediment layer. Five equally spaced cores will be collected from the length of shore sampled for resident fish. The upper layers from each core will be composited into a single sample representing the electrofishing zone.

Bedded sediment samples will be collected once in 1999 at the stations listed in **Table 3.2**, in late summer/early fall, under dry weather conditions, during the resident fish sampling.

Bedload sediment - sediment transported by a stream on or immediately above its bed - will be sampled using a bedload sampler under wet weather conditions at the 5 locations at which wet weather water samples will be collected (**Table 3.1**). Samples will be collected once during 1999, under wet weather conditions.

Settling sediment - sediment which settles from the water column to the stream or lake bed, usually due to a change in hydrodynamic conditions - will be sampled using a sediment trap. Three samples will be collected from each impoundment listed in **Table 3.1** once during 1999, during one wet weather event.

All sediment samples will be analyzed for PCB congeners and total organic carbon. Qualitative data on sediment composition, appearance, etc., will also be recorded, and sediment cores will be photographed.

3.6 Caged Fish and Caged Semipermeable Membrane Device Sampling

Caged young-of-the-year (YOY) channel catfish (*Ictalurus punctatus*) are routinely used by the MDEQ and others as indicators of bioavailable contaminant concentrations. Caged fish are superior to mobile resident fish as indicators of site-specific contaminant concentrations. Channel catfish are used rather than other species such as fathead minnows (*Pimephales promelas*) because catfish are more tolerant of poor water quality conditions.

SPMDs consist of a polyethylene membrane packet containing a small amount of the triglyceride lipid, triolein. SPMDs accumulate hydrophobic contaminants by passive diffusion, in a manner similar to many aquatic organisms, and have been used as surrogates for fish in a variety of studies. SPMDs are especially useful for determining site-specific concentrations of bioavailable contaminants in locations where aquatic organisms may not survive.

Caged channel catfish and caged SPMDs will be deployed as near as possible to ten of the eleven locations at which resident fish will be collected (**Table 3.2**). Four composite samples of whole-body channel catfish and three SPMDs will be collected at each location. Both caged fish and caged SPMDs will be left in-place for 28 days, in the late summer/early fall (**Appendices D and E**). The SPMDs will be retrieved three times during the exposure period, to clean them of biofouling organisms. At those times, water column dissolved oxygen measurements will be made to assess whether the caged fish are under stress.

Caged fish and SPMDs will be analyzed for PCB congeners, and the fish tissues will also be analyzed for total lipids. Lipid data will be used to normalize the wet weight PCB concentrations. PCB data from caged fish and SPMDs will be assessed for comparability to other sample media following the 1999 field season.

SECTION 4

Field Quality Assurance/Quality Control

4.1 Sampling Documentation - Field Notebook and Field Data Sheets

The field notebook will be a dedicated, bound notebook containing waterproof paper, and will contain an overall record of all activities performed in the field, including, but not limited to:

- Sample collection date and time
- Sampling location (GPS coordinates, plus other related observations)
- List of field personnel
- Weather conditions
- Sample types collected
- Sample identification numbers
- Other appropriate observations and comments
- Photograph frame numbers, if any are taken
- Signature of the data recorder and the designated team leader

If an error is made during notebook entry, the error will be crossed out and the correct information entered. The erroneous information will not be obliterated, and all corrections will be initialed by the data recorder and dated.

Sampling location coordinates will be measured using a geographic positioning system (GPS) where possible (i.e., where bridges or streamside vegetation do not interfere with satellite signal reception.) In addition, detailed descriptive notes of station location - position in the river channel, approximate distance to shore, proximity to local landmarks, etc. - will be recorded.

Selected field records will also be recorded on field log sheets. All field information will be recorded in waterproof ink. A copy of all field notebooks and photographs will be made available to MDEQ

4.2 Sample Handling

After collection, samples of all types will be handled using the same procedures:

- Labels will be checked for accuracy and taped to protect information.
- Chain of custody sheets will be completed using information from the field notebook.

- Sample containers will be placed on ice, in ice chests. Laboratory copies of the chain of custody forms will also be placed in the ice chests, inside zip lock bags.
- Ice chests will be sealed with tape & custody seals at opposite corners to prohibit tampering. Ice chests will be shipped overnight to the appropriate analytical laboratory.

Details of sample containers, preservation techniques, and holding time requirements are provided in Table 4.1.

Table 4.1 Sample Container, Preservation, and Holding Time Requirements

Parameter	Container	Sample Size	Preservation	Maximum Holding Time
Surface Water				
PCBs	Amber glass bottle with Teflon® lined cap	1 liter	Cool, 4 °C	Extract within 5 d of sample receipt; analyze within 30 d of extraction
TSS	Plastic	500 mL	Cool, 4 °C	7 days
Resident and Caged Fish				
PCBs	Wrap with aluminum foil and place in zip-lock bag	Minimum 20 g	Cool, 4 °C; Lab = freeze < -20 °C	Extract within 6 months of collection; analyze within 30 d of extraction Sediment (all types)
Sediment (all types)				
PCBs	Flint glass jar with Teflon® lined cap	Minimum 100 g	Cool, 4 °C	Extract within 28 d of collection; analyze within 30 d of extraction
TOC	Plastic bottle	20 g	Cool, 4 °C	28 days
Caged SPMDs				
PCBs	Metal can supplied by SPMD manufacturer	1 SPMD	Cool, 4 °C	Extract within 10 d of collection; analyze within 30 d of extraction

4.3 Sampling Equipment Decontamination

The specific procedure for decontaminating sampling equipment (Table 4.2) is described in the Field Equipment Decontamination SOP (Appendix F).

Table 4.2 Sample Collection Equipment Requiring Decontamination

Sample Type	Equipment	Decontamination Frequency
Surface Water	Filler cap	Between each sample
Sediment	Sediment homogenization equipment: bowls, spoons, etc.	Between each sample
Resident Fish	Fillet knives	Between each sample

PCBs are the primary contaminant of concern. PCBs are semivolatile organic compounds not subject to rapid photodegradation, and so precautions against volatilization or exposure to sunlight are unnecessary. In general, equipment will be cleaned using soap and water washing followed by rinsing with a polar solvent (methanol) and a nonpolar solvent (n-hexane), and finally rinsing with distilled water. The soap and water washing is intended to remove oils, etc., while the two solvents will remove PCBs and other contaminants of varying polarities.

Special effort will also be made to minimize sample contamination by keeping the sampling vessel, field vehicles, and the clothing of the field crews as clean as possible.

4.4 Quality Assurance Objectives for Measurement Data

The overall objective of a Quality Assurance Program is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting to provide results which are legally and technically defensible. This is accomplished by applying specific QA/QC procedures designed to produce analytical data of known and measurable quality.

4.4.1 Precision

Precision is the reproducibility or degree of agreement among replicate measurements of a single analyte or property. The closer the numerical values of the measurements are to each other, the more precise the measurement. The measure typically used to estimate the precision of a method is the standard error of the estimates for the least square regression line of "measured" vs. "target" concentrations.

Analytical precision will be determined through the use of matrix spikes and matrix spike duplicates for the analytical work performed. Table 4.3 describes the QA/QC precision for the Long Term Monitoring.

Table 4-3 Frequency of Field QC Sample Preparation

Sample Type	Field Duplicate	MS/MSD	Equipment Rinsate Blank
Surface Water	One Sample per Survey	Every 20th Sample	Every 10th Sample
Bedded Sediment	Every 10 th Sample	Every 20 th Sample	Every 10 th Sample (silica sand blank)

4.4.2 Accuracy

Accuracy refers to the degree of difference between measured or calculated values and the accepted reference value. The closer the numerical value of the measurement comes to the reference value, or actual concentration, the more accurate the measurement. Analytical accuracy is expressed as the percent recovery of an analyte which has been added to the environmental sample at a known concentration before analysis. For laboratory, accuracy will be determined from the results from the matrix spike analysis. Percent recovery will be determined and reported for all matrix spike samples (Table 4-3). The laboratory specific QAPP provides the equations and control limits to be used for accuracy determination.

4.4.3 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Following completion of field work and analytical testing, the percent completeness will be calculated by the following equation:

$$\text{Completeness (\%)} = \frac{(\text{number of accepted and approved values reported}) \times 100}{(\text{number of samples submitted for analysis for each parameter})}$$

The acceptable range for completeness is 95% for all parameters.

Field Completeness Requirements

Field sampling conditions are often unpredictable and nonuniform. The objective of the field sampling program is to obtain samples for analyses required, to provide sufficient sample material to complete those analyses, and to produce samples to demonstrate QC for sampling procedures, where possible. The analysis of rinsate blanks and field duplicates, where possible, will provide the basis for determining acceptance of data.

Laboratory Completeness Requirements

The analytical laboratory's goal will be to provide data meeting QC acceptance criteria for 95 percent or more of all samples tested using specified laboratory methods. Completeness of the analytical results will be determined from the data validation process. Data characterized as usable in the validation will be counted toward the specified completeness target of 95 percent; however, data classified as usable with qualifications or as unusable will not be counted toward completeness.

4.4.3 Representativeness

Representativeness expresses the degree to which data accurately and precisely represents a characteristic of the media or material being sampled.

Representativeness is a qualitative parameter, which is dependent upon the proper design of the sampling program and proper laboratory protocol. The sampling network was designed to provide data representative of conditions at the API/PC/KR site by verifying existing data and comparing measurements made from sampling period to sampling period.

Measures to Ensure Representativeness of Field Data

Representativeness will be satisfied by ensuring that proper sampling techniques are used and proper field measurement procedures are followed, as specified in the SOPs.

Measures to Ensure Representativeness of Laboratory Data

Representativeness will be satisfied by ensuring that proper analytical procedures are followed and holding times of the samples are not exceeded in the laboratory. Representativeness will also be assessed by reviewing the analysis of, rinsate blank and field duplicate samples.

4.4.4 Comparability

Comparability expresses the confidence with which one data set can be compared with another. The extent to which analytical data are comparable depends on the similarity of sampling and analytical methods. The procedures used to obtain the new analytical data are expected to provide data comparable to existing data. These new analytical data, however, may not be directly comparable to existing data because of differences in procedures and QA objectives associated with the collection of the past data over a period of several years by the PRPs consultants. All data collected in subsequent sampling efforts under this plan or its addenda, are expected to be directly comparable to the data collected in 1999.

4.5 Corrective Actions

Corrective actions may be required for two classes of problems:

- analytical and equipment problems
- noncompliance problems.

Analytical and equipment problems may occur during sampling and sample handling, sample preparation, laboratory instrumental analysis, and data review. For noncompliance problems, a formal corrective action program will be initiated as soon as the problem is identified. The person who identifies the problem is responsible for notifying the CDM Project Manager, who is in turn responsible for notifying the CDM Project Quality Assurance Officer (QAO). Implementation of corrective action will be confirmed in writing through the same channels.

Any nonconformance with the established quality control procedures in the FSP will be identified and corrected in accordance with this Field Sampling Plan. MDEQ will be notified of a nonconformance situation in as timely a manner as possible.

Corrective actions will be implemented and documented, and MDEQ Project Manager will be notified. No staff member will initiate corrective action without approval of the CDM Project Manager. If corrective actions as implemented are insufficient, work may be stopped by the CDM's QAO.

4.5.1 Field Corrective Action

Technical staff and project personnel will be responsible for reporting all suspected technical or QA nonconformances or suspected deficiencies of any field activity or issued document by reporting the situation to the CDM Project Manager or designee. This manager will be responsible for assessing the suspected problems in consultation with the CDM's QAO, and on making a decision based on the potential for the situation to impact the quality of the data. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the CDM Manager.

The CDM Project Manager will be responsible for ensuring that corrective action for nonconformances are initiated by:

- evaluating all reported nonconformances
- controlling additional work on nonconforming items
- determining disposition or action to be taken
- maintaining a log of nonconformances
- reviewing nonconformance reports and corrective actions taken

- ensuring nonconformance reports are included in the project files.

If appropriate, the CDM Field Manager will ensure that no additional field work that is dependent on the nonconforming activity is performed until the corrective actions are completed.

Corrective action for field measurements may include:

- repeat the measurement to check the error
- check for all proper adjustments for ambient conditions such as temperature
- check the batteries
- check the calibration
- replace the instrument or measurement devices
- stop work (if necessary).

The CDM Project Manager or his designee is responsible for all project activities. In this role, CDM's Project Manager at times is required to adjust the project programs to accommodate project specific needs. When it becomes necessary to modify a program, the responsible person notifies the manager of the anticipated change and implements the necessary changes after obtaining the approval of the CDM Project Manager. The change in the program will be documented on a field change request that will be signed by the initiators.

Field change requests will be numbered sequentially and attached to the file copy of the affected project document. The CDM Project Manager must approve the change in writing or verbally prior to field implementation, if feasible.

The CDM Project Manager is responsible for controlling, tracking, and implementing the identified changes and notifying the MDEQ Project Manager. Reports on all changes will be distributed to all affected parties.

4.5.2 Laboratory Corrective Action

Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. The investigative action taken is somewhat dependent on the analysis and the event.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy
- blanks contain target analytes above acceptable levels

- undesirable trends are detected in spike recoveries or RPD between duplicates
- there are unusual changes in detection limits
- deficiencies are detected by the QA personnel during internal or external audits or from the results or performance evaluation samples
- inquiries concerning data quality are received.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the Technical Director, Laboratory Director and/or Laboratory QA Manager for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the CDM's QAO.

Precision and accuracy will be regularly tracked by the analytical staff to determine unacceptable results and to evaluate and implement corrective actions. Laboratory supervisors and QA/QC staff will evaluate analytical data against the appropriate quality control limits. Corrective actions may include, but are not limited to, recalibration of instruments using freshly prepared calibration standards; replacement of lots of solvent or other reagents that give unacceptable blank values; additional training of laboratory personnel; or reassignment, if necessary. Corrective actions in many cases may have to be defined as the need arises;

If substantial corrective action is required or if serious QA problems are encountered, CDM's QAO will be notified by the Laboratory QA Manager by phone and in writing as soon as possible. All corrective actions will be implemented and documented after notification of the CDM's QAO and the MDEQ Project Manager.

For nonconformance problems, a formal corrective action process is initiated as soon as the problem is identified. The individual who identifies the problem is responsible for initiating the documentation. The nonconformance is reported to the appropriate supervisor, who will complete and sign a nonconformance memorandum form and notify the Laboratory QA Manager. The Laboratory QA Manager is responsible for tracking the status of the nonconformance memos. Implementation of corrective action will be confirmed in writing through the same channels. Any nonconformance memo that has been written against a specific project is filed with the project file and is noted in the case narrative of the Certificate of Analysis or Data Package. Implementation or corrective action within the laboratories is focused through the Laboratory Project Managers' office.

For additional information on laboratory corrective action, refer to the Laboratory QAAP.

4.5.3 Corrective Action During Data Validation and Data Assessment

Any nonconformance with field or laboratory QA objectives noted during data validation and assessment will be referred to the Laboratory AM Manager and Laboratory Project Manager, respectively, for implementation of appropriate corrective action after review with the CDM Project Manager and CDM's QAO. Appropriate corrective action may include reanalysis or resampling.

4.6 Quality Assurance Responsibilities

4.6.1 CDM Project Quality Assurance Officer

The CDM Project Quality Assurance Officer is responsible for the overall control of all project related QA/QC activities. She/he has the authority and responsibility to identify quality problems; initiate, recommend, or provide corrective actions; and verify the implementation of the corrective actions. The CDM Project Quality Assurance Officer will also:

- Provide an independent QA function
- Review and approve QA plans and procedures
- Provide QA assistance to CDM/subcontractor staff
- Request action to be taken and/or review actions which have been taken should there be a quality control problems

4.6.2 CDM Laboratory Quality Assurance Officer

The CDM Laboratory Quality Assurance Officer will be responsible for providing an independent review of all laboratory data packages. The QA officer will report directly to CDM's Project Quality Assurance Officer and the Project Manager. CDM's QAO is Wendy Dewar and reports to CDM's Project Manager Ronald French. CDM's Laboratory Quality Assurance Officer is Todd Burgesser.

4.7 Photograph Log

A log will be kept of all photographs taken during the study. The log will record the data, time, frame and roll number, and the photographer. The serial number of the camera and lens used to take the pictures will also be recorded in the log book. A copy of all field notebooks and photographs will be made available to MDEQ for project filing.

Section 5

Data Reduction, Validation, and Reporting

5.0 Introduction

Since the data generated by this monitoring program, especially the congener-specific PCB analyses, will be voluminous, it is essential that the data management procedures maintain the quality and integrity of the data, and efficiently store, screen, and retrieve them for subsequent analysis and interpretation. An overview of these procedures is provided below.

5.1 Data Reduction

Data reduction is an integral part of any field investigation. This process often includes applications of methods, calculations, and computer programs. Documentation of all methods, calculations and data must be sufficient to allow a technically qualified person to review and decipher calculations and data to verify results.

5.1.1 Field Data

Field data will be recorded in field books all field generated data will be retained by CDM in permanent files in the CDM Detroit office (One Woodward Avenue, Ste.1500, Detroit, Michigan). One copy of the field data will be delivered to the MDEQ project manager on a yearly basis. The CDM Site Manager will be responsible for organizing the field data records and conveying them to the CDM Project Manager, who will arrange for permanent filing of the records. The Site Manager will be responsible for checking all data, calculations and methods collected by other field personnel.

5.1.2 Sample Numbering

Efficient and accurate data handling begins with properly labeling the samples in the field. All samples will be assigned a unique ~~4~~⁵-digit sample number. This sample number will be assigned to both abiotic and biotic media collected from the API/PC/KR site. The sample designations will be as follows:

aa-bbb-c-dd

where aa=sample matrix type, bbb=station number, c=location on transect (if any), and dd=multiple indicator (for blanks, duplicates, etc.).

Table 5-1. Sample Matrix Abbreviations.

Matrix Abbreviation	Explanation
SW	Surface water
AF	Adult fish
YF	Yearling fish
SP	Semipermeable membrane device
BS	Bedded sediment
BL	Bedload sediment
SS	Settling sediment

This sample number will follow the sample and its data through the entire data management system. Additional data associated with each sample will include:

- Sample collection time (military time)
- Sampling station location (latitude and longitude and/or river mile)
- Samplers identity
- Miscellaneous remarks and comments

5.1.3 Database

The database software used in this study will be the National Oceanic and Atmospheric Administration's (NOAA's) Query Manager 1.4 with MARPLOT™. This database was developed by NOAA's Hazardous Materials Response and Assessment Division, and will be used to manipulate the data for importing into mapping and graphical software packages, and for report preparation.

The NOAA has developed combined database and mapping projects using both MARPLOT and GIS ArcView graphical display capabilities to show the spatial relationships of sediment contaminant and toxicity data, natural resources, and habitat restoration projects; in the context of a watershed's features and land uses. Projects collect and summarize available information on chemical contamination of sediment and biota, and biological effects in the watershed of concern. Graphical displays facilitate a watershed approach to assessment and remediation.

Query Manager

Applicable to all NOAA Watershed Projects and other similar projects, the cross-platform Query Manager is a useful interactive system for displaying different types of data on maps and for providing a large amount of data in an easy-to-use and standardized format. Query Manager is based on a standardized database structure for all watershed projects, allowing users to select from a menu of database queries on sediment chemistry (surface and subsurface), sediment toxicity, and tissue chemistry, and automatically displays the results on a map in MARPLOT7 or saves the extracted data in an easy-to-use format for use with other applications such as ArcView.

Software Packages

Software packages used to work with the data include:

Microsoft Access and Excel spreadsheet software
ArcView and ArcInfo geographic information system mapping software

5.2 Data Validation

5.2.1 Procedures Used to Validate Field Data

Field data generated in accordance with this workplan will include temperature (water and air), water pH and specific conductance. All field data will be validated by review of the field notes and equipment calibration logs to check that all procedures have been performed and the information has been recorded appropriately. This documentation will be considered sufficient to provide that proper procedures have been followed during the field investigations.

The CDM Project Manager will be responsible for periodically checking field sampling to verify that field measurements and sampling protocols have been observed and adhered to: The checks will include:

- Use of approved procedures
- Date/Time sampled
- Preservation method
- Chain-of-Custody protocols
- Field log books

5.2.2 Procedures Used to Validate Laboratory Data

Data validation provides an independent third party review in detail of written reports of laboratory analytical results and supporting QA/QC data to assess usability of the data for evaluating the effectiveness of a remedial alternative(s). For the first year of sampling, 100% of the data will be validated in accordance with USEPA National Functional Guidelines for data quality level objective levels IV and V. Based on the results from the first year of sampling and validation, future sampling events may be subject to less stringent validation procedures, as directed by MDEQ. Appendix B contains the Standard Operating Procedures and all checklists for the State Designated Laboratory. The third party data validator will be CDM's Laboratory Quality Assurance Officer.

Procedures used to validate data integrity are:

- Completeness of laboratory deliverable data package

- Traceability of the sample from receipt to data reporting by the use of Analysis Request and Chain-of-Custody forms and unique sample numbers
- Conditions of sample upon receipt by the lab compared to shipping criteria for that type sample (e.g., refrigeration)
- Maintenance of holding time requirements
- Comparison of initial and continuing calibration results to method calibration criteria. The initial calibration and daily calibration checks must meet the criteria specified. If these criteria are not met, the sample will be reanalyzed or the data will be flagged
- MS/MD results for PCBs and duplicate results for inorganics. These results will be compared to the appropriate method criteria. Data not meeting the listed quality control criteria will be flagged
- Evaluation of method blanks. Method blank results will be compared to the appropriate method criteria. If the method blank results do not meet the required criteria stated in the appropriate method, all affected samples will be reextracted and reanalyzed

Completeness checks will be made on all data to ascertain that deliverables specified in the work plan are present. Deliverables include sample chain-of-custody forms, analytical results, designated QC summaries and supporting raw data from instrument printouts (e.g., chromatograms). The CDM Laboratory QA reviewer will make sure all required items are present and request the laboratory to obtain the missing deliverables.

5.3 Laboratory Data Reporting

The State Designated Laboratory will provide the following information to form a data report package:

- Date of issue
- Laboratory analysis performed
- Any deviations from the stated analytical method
- Laboratory batch number
- Number of samples and the sample matrices

- Reference to the quality control procedures performed for the specific methods used, including the reference to the acceptance criteria used
- Contents of the laboratory report
- Project name and number
- State of the sample received (e.g., container found open)
- Whether sample holding times were met and identification of those samples for which they were not met
- Any observations that may have had an impact on the analyses, and corrective actions taken
- All chromatograms for PCB congeners
- All chemical data packages (e.g., instrument logs, calibration results, results from matrix spikes, MSDs, etc.)
- Laboratory quality control checks that did not meet the project/laboratory criteria
- The Laboratory Project Manager's signature approving the issuance of the data package

A copy of the Analysis Request and the Chain-of-Custody form with all signatures will accompany each data package.

Section 6

Analytical Methods Summary

6.1 Conventional Parameters

Total suspended solids in surface water will be analyzed by EPA Method 160.2 or an equivalent method. Total organic carbon in sediment will be analyzed by EPA Method 9060 or an equivalent method. Fish tissue lipids will be measured using a gravimetric technique, using an aliquot of the sample extract.

6.2 Polychlorinated Biphenyls (PCBs)

PCBs will be analyzed by congeners and total PCBs where applicable. This means that the laboratory will be required to use a capillary-type GC column, which provides good resolution of most (if not all) individual PCB congeners, and to quantitate separately the PCB concentration associated with each GC peak. In most cases, each individual peak will represent a single congener. However, some peaks will represent coeluting congeners, usually no more than two or three. The laboratory will be required to resolve complex PCB mixtures into at least 117 peaks representing the congeners present in commonly used Aroclors, as well as their dechlorinated degradation products.

The purposes for using congener analysis, rather than Aroclor analysis, are to: (1) obtain lower detection limits for total PCBs; (2) obtain more reliable data on total PCBs, especially for biological samples; (3) provide data which are more suitable for use in assessments of PCB fate and transport, including the presence of any natural attenuation; and (4) provide the equivalent of a "fingerprint" with which to evaluate the possibility of alternate PCB sources within - or discharging to - the Kalamazoo River after remediation

There are currently no standard methods for PCB congener analysis, and thus the laboratories using this approach have commonly developed their own in-house protocol. In selecting a laboratory for the congener analyses, MDEQ/CDM will review the existing in-house method/protocols to insure they would produce data compatible with this program's goals. Some of the initial sample preparation steps, and some GC features, are expected to be similar to those specified in EPA's SW-846 ("Test Methods for Evaluating Solid Waste"), e.g., those under Method 8082: "Polychlorinated Biphenyls by Gas Chromatography." A typical process could involve initial extraction with methylene chloride (water matrix) or 1:1 hexane-acetone (sediment matrix) and sample cleanup (e.g., via sulfuric acid addition, florisil treatment and sulfur removal) prior to GC injection. The GC system may involve single or dual column operation, the latter used either for

confirmation and/or for resolution of peaks coeluting on the first column. System calibration will be with a mixture - of known concentrations - of each of the congeners being identified and quantified. Laboratory QC practices will involve the use of internal standards, lab blanks, blank spikes and matrix spike/matrix spike duplicates. PCB detection may be by Electron Capture Detector (ECD), Electrolytic Conductivity Detectors (ELCD) or Mass Spectrometry (MS). The selected laboratory will be required to submit backup information relating to the analytical protocol, peak quantitation and QC procedures.

Table 6.1 Summary of Analytical Methods

Analyte	Method
PCBs	Laboratory Specific
Tissue Lipds	SW-846(3540)
Total Organic Carbon	Lloyd Kahn or equivalent
Total Suspended Solids	Standard Method 208D
Water Temperature	Thermometer

SECTION 7 Project Schedule

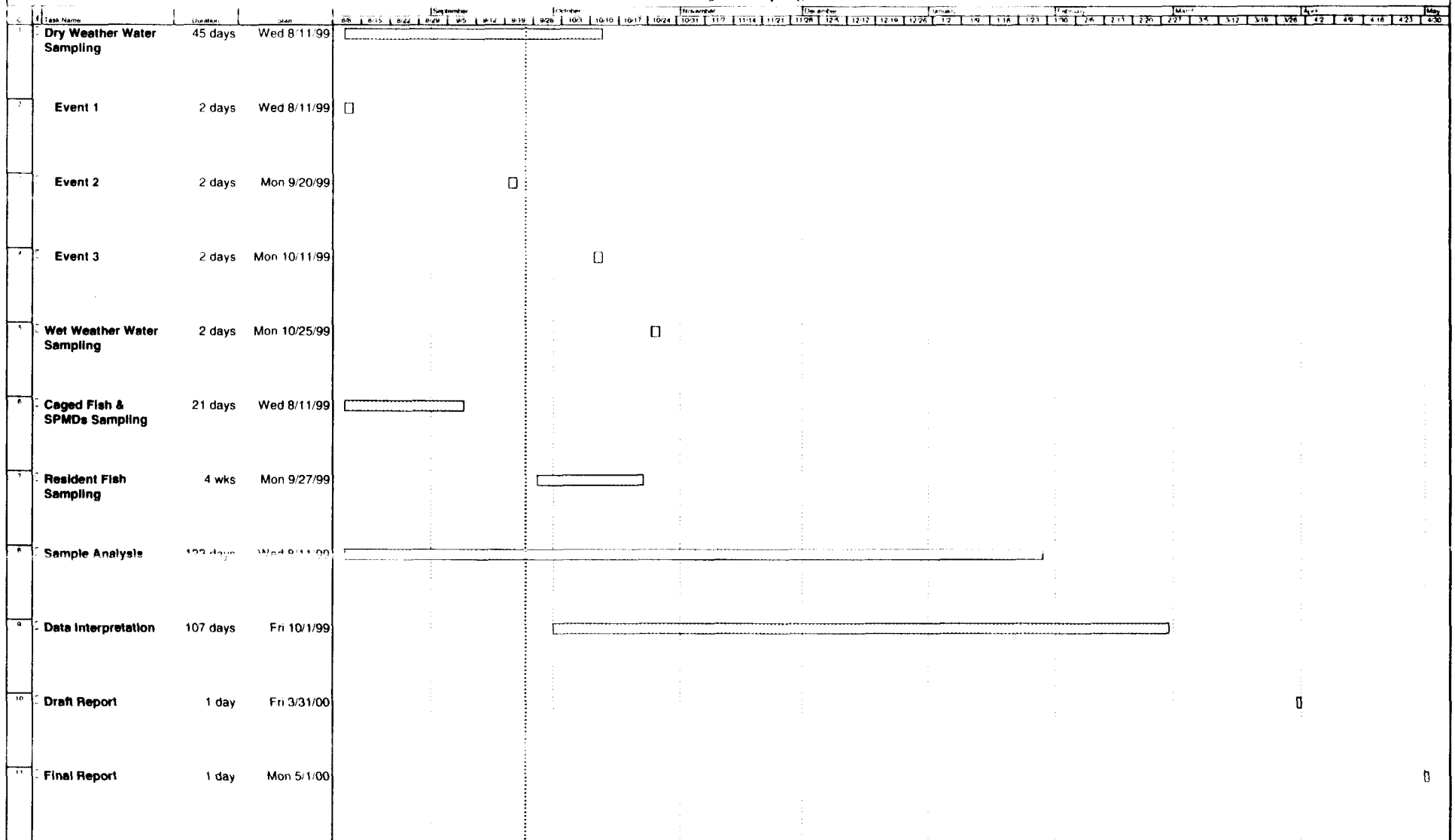
7.1 Project Schedule

Following acceptance of the Field Sampling Plan by the Michigan Department of Environmental Quality, CDM will initiate field planning and sampling activities. 1999 sampling activities will be conducted according to the schedule shown in Figure 7.1.

7.2 Project Deliverables

Upon receipt of the laboratory analytical results, CDM will prepare a monitoring report outlining all field and analytical activities. CDM will submit three draft copies of the report for MDEQ review. Upon receipt of comments from MDEQ, CDM will prepare 20 final copies for MDEQ disposition.

FIGURE 7-1. Project Sampling Schedule



Appendices SOPs

Appendix A
Collection of Depth-Integrated Surface
Water Samples for Organic Contaminant
Analysis

STANDARD OPERATING PROCEDURE

COLLECTION OF DEPTH-INTEGRATED SURFACE WATER SAMPLES FOR ORGANIC CONTAMINANT ANALYSIS

1.0 SCOPE AND APPLICATION

This standard operating procedure describes collecting a small-volume (1 liter) depth-integrated surface water sample, for organic contaminant analysis. It is applicable to any surface water; streams, lakes, impoundments, wetlands, etc.

This procedure was originally developed by the U.S. EPA – Large Lakes Research Station (Grosse Ile, MI) for use on the EPA-sponsored Upper Great Lakes Connecting Channel Study (UGLCCS; U.S. EPA, 1988).

2.0 METHOD SUMMARY

A clean 1-liter amber glass sample bottle is equipped with a “filler cap” (described in Section 3), attached to a weighted harness, submerged into the water body to be sampled, and raised and lowered from near the surface to near the bottom until the bottle is full.

3.0 PROCEDURE

3.1 Materials and Supplies

- 1-liter amber glass bottles; certified pre-cleaned
- filler caps (described below)
- bottle harness and rope (described below)
- appropriate sampling vessel (boat, canoe), if sampling from on the water
- ice chests
- decontamination supplies (see the Field Equipment Decontamination SOP)
- appropriate equipment for establishing station position (e.g., global positioning system, etc.; project-specific)
- appropriate field log book (project-specific)

Filler Cap Description

The filler cap consists of a normal bottle cap, with a Teflon® liner, into which two holes have been drilled. Into these two holes should be placed two lengths of stiff Teflon® tubing, both 3/16” ID, one ¾” long and the other 1 ¼” long.

Bottle Harness Description

The bottle harness can take a variety of shapes:

- A piece of 6"-tall, 6"-diameter plastic pipe equipped with an end cap, and a foam insert appropriate to securely hold the sample bottle inside the pipe. This design was used successfully on the U.S. EPA-funded Rouge River National Wet Weather Demonstration Project (Detroit, MI).
- A metal base plate (aluminum or stainless steel) weighted so as to submerge the sampler, attached to an upper plate by threaded metal rods. The upper plate should have a cutout appropriate to allow the sample bottle neck to protrude above it. The upper plate is secured to the threaded rods with wing nuts, holding the sample bottle securely between the upper and lower plates. This design was used successfully in the UGLCCS study.

The harness should be attached to an appropriate length of rope (1/4" nylon or polypropylene rope recommended), which must be long enough to allow the bottle/harness unit to be lowered to within a foot or two of the bottom. Between stations, the harness and rope can be conveniently stored in a plastic bucket.

This harness can also be used for collecting water samples for other analyses (total suspended solids, nutrients, etc.) by substituting the appropriate sample bottle for the glass 1-L bottle.

3.2 Procedure

[Note: steps marked with "***" denote general descriptions of activities that will vary from project to project, the details of which are described in detail in the project-specific Field Sampling Plan.]

1. Establish position on station and record location in the field notebook.** Appropriate procedures could include use of a global positioning system, triangulation on local landmarks, careful notations on a detailed map, etc.
2. Label bottle with station number (or other identifier), date, and time, and wrap label with clear waterproof tape.
3. Place a clean filler cap on a pre-cleaned 1-liter amber glass bottle. Take care to keep the bottle's original cap clean; wrapping it in solvent-rinsed aluminum foil is recommended.
4. Secure the bottle in the bottle harness.
5. Place the bottle into the water column, and begin raising and lowering the bottle and harness up and down through the water column until the bottle is full. This will typically take 2 to 4 minutes. Use caution not to let the bottle come in contact with the sediment, or sample the surface water film; see Section 4.
6. When the sample bottle is full, remove it from the harness and replace the filler cap with the original bottle cap.
7. Place the bottle in a zip lock bag, and place into an ice chest. Place on ice until transfer to the analytical laboratory.

4.0 FIELD QA/QC PROCEDURES

1. To minimize sample contamination, take care not to allow the sample bottle and harness to come into contact with the bottom sediment, or to sample the surface film.
2. Details of the field QA/QC procedures will be project-specific and are described in the project Field Sampling Plan. Typical field QA/QC operations include:
 - Collection of duplicate water samples and matrix spike/matrix spike duplicates, at regular intervals (a certain number per day or per water body sampled, or 1 per 10 or 20 samples)
 - Equipment rinsate blanks, of the filler caps.
3. Between samples, filler caps should be cleaned by:
 - Washing with soap and water
 - Rinsing once with tap water
 - Rinsing once with methanol
 - Rinsing once with n-hexane
 - Rinsing once with distilled water

If not reused immediately, filler caps should be wrapped in solvent-rinsed aluminum foil.

5.0 HEALTH AND SAFETY

Normal water safety precautions must always be observed, including wearing life vests, taking care to avoid hypothermia or heat stroke, etc.

6.0 REFERENCES CITED

U.S. EPA. 1988. *Upper Great Lakes Connecting Channels Study – Volume II – Final Report*. U.S. EPA Great Lakes National Program Office, Chicago, IL. 626 pp.

Appendix B
Residential Fish Collection by Boat-Mounted
Electrofishing and Sample Processing for
Organic Contaminant Analysis

STANDARD OPERATING PROCEDURE

RESIDENT FISH COLLECTION BY BOAT-MOUNTED ELECTROFISHING AND SAMPLE PROCESSING FOR ORGANIC CONTAMINANT ANALYSIS

1.0 OVERVIEW AND APPLICATION

This standard operating procedure describes field procedures for collecting resident fish from rivers, lakes, etc., and sample processing procedures for preparing fish carcass or fillet samples for organic contaminant analyses.

2.0 METHOD SUMMARY

Resident fish are collected by electrofishing from a boat, and processed for organic contaminant analysis as either fillet or whole carcass samples.

3.0 PROCEDURE

3.1 List of Equipment and Supplies

- Electrofishing unit. This will consist of:
 - electrofishing boat and trailer
 - electrodes
 - gasoline powered generator
 - control equipment to regulate voltage and current to the electrodes
 - wiring to provide safe transmission of current under operating conditions
 - switching, including a switch that keeps the circuit open unless actively and continuously closed (i.e., the “dead-man’s switch”)
 - dip nets with handles of non-conductive material
 - personal safety equipment including life vests, footwear with non-conductive soles, and non-conducting gloves
- Global positioning system, or mechanism for recording sampling location position
- Fillet knives
- Solvent-rinsed heavy duty aluminum foil
- Zip lock freezer bags
- Ice chests and ice
- Fish measuring board or tape measure
- Appropriate scale for weighing fish
- Appropriate forms and/or field notebook
- Laboratory chain of custody forms
- Tap water
- Methanol
- N-hexane
- Waste containers for methanol and hexane

- Distilled water
- Camera and film
- Current scientific fish collector's permit

3.2 Procedure

3.2.1 Setup and Checkout

The field crew will set up and test the electroshocking equipment in accordance with the procedures specified in the operating manual for the unit(s) employed at the site.

3.2.2 Field Crew Responsibilities

The field crew will consist of the Crew Leader and the Crew Member(s). The Crew Leader will be responsible for :

- Control of the boat
- Operation of the control equipment and generator.

The Crew Member(s) will be responsible for:

- Control of the dead-man's switch
- Capturing the fish

Both the Crew Leader and the Crew Member(s) will be responsible for preparing the fish samples for shipment to the laboratory.

3.3 Electrofishing Operation

1. Apply current to the water by closure of the dead-man's switch while the generator and control equipment are operative.
2. Collect fish using dip nets when seen, and transfer to on-board live well (or other containers filled with water).
3. (Optional) – Use a geographic positioning system to record position and boat track while sampling.

3.4 Fish Sample Preparation

1. Return the collected fish to shore.
2. Identify the fish to species.
3. Measure fish length and weight, and record in field notebook and/or field data sheets.
4. Photograph the fish, including a small tag on which is written the sample number.
5. If whole fish are to be processed, proceed to step 6. If fish fillets are to be collected, first kill each fish by severing its spinal cord using a fillet knife, and then remove the fillets from the carcass. If necessary, remove skin from each fillet.

6. Wrap the whole fish or fillets from an appropriate number of fish in solvent-rinsed aluminum foil (dull side in), and label appropriately (station number or other identifier, date, fish species, number of fish or fillets). Record this information in the field notebook and/or field data sheets.
7. Place the aluminum foil-wrapped samples into similarly-labeled zip lock bags.
8. Place the samples into ice chests, and transfer to the analytical laboratory as quickly as possible.

4.0 FIELD QA/QC

1. Details of the field QA/QC procedures will be project-specific and are described in the project Field Sampling Plan. Typical field QA/QC operations include:

- Collection of duplicate fish samples, at regular intervals (a certain number per day or per water body sampled, or 1 per 10 or 20 samples)
- Equipment rinsate blanks, of the fillet knives and aluminum foil.

2. Between samples, fillet knives (if used) should be cleaned by:

- Washing with soap and water
- Rinsing once with tap water
- Rinsing once with methanol
- Rinsing once with n-hexane
- Rinsing once with distilled water

If not reused immediately, fillet knives should be wrapped in solvent-rinsed aluminum foil.

5.0 HEALTH AND SAFETY

1. Non-conducting boot soles and gloves, worn in conjunction with proper operation of the dead-man's switch, will protect the field crew from shocking hazard during this operation.
2. Normal water safety precautions must always be observed, including wearing life vests, taking care to avoid hypothermia or heat stroke, etc.
3. Sampling will be temporarily halted when any persons, pets, or livestock are observed in the water or on the shore within approximately 40 feet of the electroshocking unit.
4. Sampling will also be halted when any other boats are observed within 50 yards of the electroshocking boat.

Revised
GLEAS Procedure #31

Fish Contaminant Monitoring Program
Fish Collection and Processing Procedure

Introduction

The purpose of the Fish Contaminant Monitoring Program (FCMP) is to quantitatively assess the degree of chemical contamination in fish from waters throughout the state. This procedure describes the collection and processing techniques for fish samples to be obtained for contaminant analysis.

Pre-Collection

Staff should maintain a field notebook containing the following information (as a minimum):

- This Procedure
- Fisheries Division Contact List (District Offices and Fish Stations)
- Blank Field Data Sheets
- Current Michigan Fishing Guide

Staff with the responsibility for a specified site will be provided with a Fish Collection Assignment Sheet (Attachment 1) by the FCMP Specialist. The assignment sheet identifies the waterbody, location, contact people, id#, species (number and size ranges) and processing instructions. Staff should select appropriate fish sampling techniques and collection times after consultation with the appropriate District Fisheries Biologist. Staff collecting fish samples must have Cultural and Scientific Fish Collectors Permits which are issued by Fisheries Division.

The FCMP and biosurvey (stream shocker and backpack shocker sections) checklists can be used to identify equipment and supplies needed (Attachments 2 and 3). Vehicles, boats and major equipment (shockers, nets, etc.) must be signed out on the field calendar.

The appropriate Fisheries Division Office and Conservation Officers must be notified by the GLEAS staff assigned to a site prior to the fish collections. The names and phone numbers of the people to be contacted will be provided on the Fish Collection Assignment Sheet.

Fish Collections

Most fish will be collected by GLEAS and Fisheries Division staff using electrofishing equipment or nets. Since the desired species and size ranges are sometimes not found, the Fish Collection Assignment Sheet will indicate if substitutions can be made. Substitutions will be pursuant to the predator and bottom feeder preference lists (Attachment 4). Species substitutions can not be made for trend monitoring collections. Size ranges are generally goals and should be met as closely as possible unless noted otherwise on the Fish Collection Assignment Sheet.

The minimum safety training requirements for collection staff are: CPR, First Aid, Boating Safety and Water Safety. GLEAS staff should follow electrofishing safety procedures (GLEAS Procedure #48) and other appropriate safety procedures and requirements included in the Division Safety and Health Manual.

For fish collected for composite samples, when possible the length of the smallest fish should be within 90% of the largest fish.

Once fish are collected, they should be placed on ice and processed on-site or transported to the Filley Street facility. Special sample processing and handling procedures may be necessary if chain-of-custody needs to be maintained and will be determined on a case-by-case basis. The Fish Collection Assignment Sheet will indicate if chain-of-custody needs to be maintained.

If the fish are not going to be processed on-site, then they should be:

- 1) placed in plastic bags (GLEC bags or other large garbage bags) keeping different species separate and keeping the bags under 30 lbs. each;
- 2) labelled with the waterbody, location, date and species;
- 3) placed in one of the "Fish to be Processed" freezers; and
- 4) recorded on the Freezer Log Sheet (Attachment 5).

Staff should then notify the FCMP Specialist of the number and species of fish collected.

Fish Processing

The supplies needed to process fish are listed in the FCMP Check List (Attachment 2). Fresh fish should be sorted by species and kept on ice in a shady location until processing.

To thaw frozen fish for processing:

- 1) pull fish from Filley Street freezers the afternoon before the day they are to be processed;
- 2) place the fish in tubs/coolers or on clean plastic bags in the truck well, keeping sites and species separate;
- 3) take care to avoid contaminating other equipment, especially wooden materials, which are stored in the truck well;
- 4) try to separate the fish from each other as much as possible to facilitate thawing; and
- 5) keep the doors to the truck well closed and the fan on.

The following steps should be followed for processing fish:

1. If processing at the Filley Street Facility, keep the fan on and the interior doors to the truck well closed.
2. Rinse fillet board, cleaning table and knives with water.
3. Record site information, sample#, species name, length (cms), weight (gms), sex and sample type on the FCMP Data Sheet (Attachment 6).
4. The Fish Collection Assignment Sheet will identify the appropriate recording procedure for lengths and weights for composite samples. Generally, for smaller fish species (smelt, alewife, etc.) including caged fish study samples, a range for the lengths and a total weight for the composite will be adequate. In these cases, the number of fish in the composite should be noted under the comments section of the FCMP Data Sheet. For larger fish species the length and weight of each fish in the composite is generally recorded. Attachment 6 shows an example of the data recorded for each case.
5. The comments section of the FCMP Data Sheet should be used to record the following types of information: collection date (native fish not all collected on the same date and caged fish samples), anomalies such as tumors or lamprey marks, fin clips (see Attachment 7 for fin clip codes), sample identification information for split samples...etc.
6. If instructed to collect scale/spine/otolith samples for aging follow the guidance provided in Attachment 8. This will generally only apply to trend monitoring samples.
7. If fish are to be processed whole proceed to step 10.
8. Starting with the species expected to be least contaminated (i.e. panfish) and working from the smallest to the largest specimen, process according to the Standard Edible Portion list (Attachment 9). Fillet specimen according to the figure in Attachment 10. Staff will be trained on appropriate fillet techniques.
9. Waste materials should be placed in a trash container lined with garbage bags. Thin garbage bags should be tripled, while thicker bags (i.e. garbage disposal bags) don't need to be. Bags should weigh no more than 30 lbs. Waste bags should be placed in the "Guts" freezer at the Filley Street facility unless they can be disposed of properly on-site.
10. Between each fish, the cutting board and knife(s) should be rinsed with water.

11. Wrap whole fish or edible portion sample in aluminum foil with dull side to fish. Secure package with 2" masking tape by taping lengthwise around the package along aluminum foil seam. Each fish in a composite sample must be wrapped and labeled individually, unless otherwise indicated on the Fish Collection Assignment Sheet.
12. Label each package with the following information on the masking tape using a waterproof marker:
 - date
 - waterbody
 - species
 - sample id#
 - composite number (composite samples only)
13. Place each aluminum foil package in a separate clear plastic bag (1 quart or 1 gallon zip-lock bag, or large GLEC bag; depending on size). However, for composite samples more than one package can go in the same bag.
14. Label the plastic bag with the sample id# using a waterproof marker. If more than 1 plastic bag is necessary per sample (i.e. composite samples) mark each bag with the same sample id# and label 1 of 3, 2 of 3, etc.
15. Place all the bags from a given site id# in a large plastic bag (GLEC or other garbage bag) not to exceed 30 lbs. and label bag with site id# and waterbody. If more than 1 bag is required for a given site id#, also label the bags 1 of 3, 2 of 3, etc.
16. Samples should be kept on ice until they are placed in the "Processed Fish" freezers at Filley Street and recorded on the Freezer Log.
17. Clean all of the processing equipment and return it to its proper place. If processing at the Filley Street facility, rinse floors with diluted chlorine bleach.

Post Processing

Laboratory Analysis Request Forms (Attachment 11) should be filled out and turned into the FCMP Specialist along with the completed Field Data Sheets. The forms will be maintained in the FCMP site files.

Aquatic Toxicity Laboratory staff will be responsible for transferring the bags from the guts freezer to dumpster on the morning of garbage pick-up days (currently Tuesdays).

The FCMP specialist will coordinate delivery of the fish samples to the MDPH for analysis. Aquatic Toxicity Laboratory staff will assist in the delivery.

Approved:

Date 9/13/99

Attachment 1

I.D. # _____

ASSIGNED TO: _____

WATER BODY: _____

LOCATION: _____

FUNDING SOURCE: _____

FISH DIVISION CONTACT: _____ PHONE # _____

SPECIES TO BE COLLECTED	LENGTH (INCHES)	NUMBER

SPECIAL INSTRUCTIONS:

****You can substitute another species using preference lists.**

Attachment 2

FCMP CHECK LISTBoat

Boat Plug _____
Oars _____
Anchor & Rope _____
Motor _____
Gas & Oil _____
Cushions _____
Life Jackets _____
Hitch Lock & Key _____

Fish Processing Tools

Scale & Tripod _____
Measuring Board _____
GLECS Plastic Bags _____
Ziploc Bags _____
 Gallon _____
 Quart _____
Trash Bags _____
Trash Can _____
Table _____
Tape _____
Tubs _____
Scale Envelopes _____
Pens & Markers _____
Fillet Knives & Steel _____
Labels _____
Aluminum Foil _____
Sharpening Stone _____
Wash Brush _____
Cutting Board _____
Fish Data Field Sheets _____
Fish Collection Notebook _____

Fishing Gear

Fyke/Hoop/Trap/Gill Nets _____
Net Anchors w/Ropes _____
Net Floats w/Ropes _____
Seine _____
Waders/Hip Boots/ _____
 Knee Boots _____
Gill Net Picks _____

Miscellaneous

Fathometer (connectors & _____
 battery _____
Maps _____
Cooler(s)/Ice _____
Camera & Film _____
Mosquito Repellant _____
Fish Finder/Depth Finder _____
Global Positioning System _____
Sun Block/Sunglasses _____
Raingear _____
Collectors Permit _____

Attachment 3

Stream Shocker

Sport Yak w/Bottom Board _____
Control Box _____
Ground (Floating) _____
Probes _____
Safety Switch _____
Generator _____
Gas _____
Rubber Gloves _____
Nets _____
Collection Tub _____

Clothing

Waders/Hip & Knee Boots _____
Rain Gear _____
Socks _____
Hat _____

Miscellaneous

Measuring Tape _____
Camera & Film _____
Polarized Sunglasses _____
Mosquito Repellant _____
Sunblock/Sunglasses _____
Maps _____
Sample Jars/Bottles _____
Cooler/Ice _____
Stainless Steel Bowl/ _____
Spoon (Sediment Samples) _____
Data Forms _____
Preservative Kits _____
Global Positioning _____
System _____
Business Cards _____

Backpack Shocker

Backpack Shocker _____
Charged Batteries _____
Probes _____
Ground (Floating) _____
Nets _____

Fish & Benthos Processing Gear

Measuring Board _____
Porcelain Pan _____
Bucket _____
Sieve Bucket _____
Aquarium Net _____
Tweezers/Forceps _____
Eye Dropper _____
Tubs _____
Dip Nets _____
Vials _____
Station Cards _____
Pens _____
Clipboard & Paper _____
Alcohol _____
Formalin _____
Thermometer _____
Hand Lens _____

Attachment 4

PREDATOR PREFERENCE LIST

When predator is indicated, please apply the following guidance. Our first preference is to substitute a top line predator from Group 1. If we are unable to obtain a species from Group 1, then a Group 2 species may be substituted. Size ranges are goals.

	<u>Species</u>	<u>#of Fish</u>	<u>Size Ranges</u>
Group 1:	Walleye	5	15" – 18"
		5	19 +
	Northern Pike	5	24" – 25"
		5	26" +
	Smallmouth Bass	5	14" – 15"
		5	16" +
	Largemouth Bass	5	14" – 15"
		5	16" +
Group 2:	Yellow Perch	10	9" +
	Black Crappie	10	9" +
	Rock Bass	10	9" +

Bottom Feeder Preference List

When bottom feeder is indicated, carp is always the preferred species. If carp are not available, one of the other species listed below can be substituted.

<u>Species</u>	<u># of Fish</u>	<u>Size Range</u>
Carp	5	18" – 22"
	5	23 +
Sucker sp.*	10	12" +
Channel Catfish	10	12" +
Bullhead sp.*		

Please do not mix different species in a sample (i.e. use 10 White Sucker or 10 Redhorse Sucker)

Attachment 5

Freezer Number[illegible]

Township _____ Range _____ Action _____

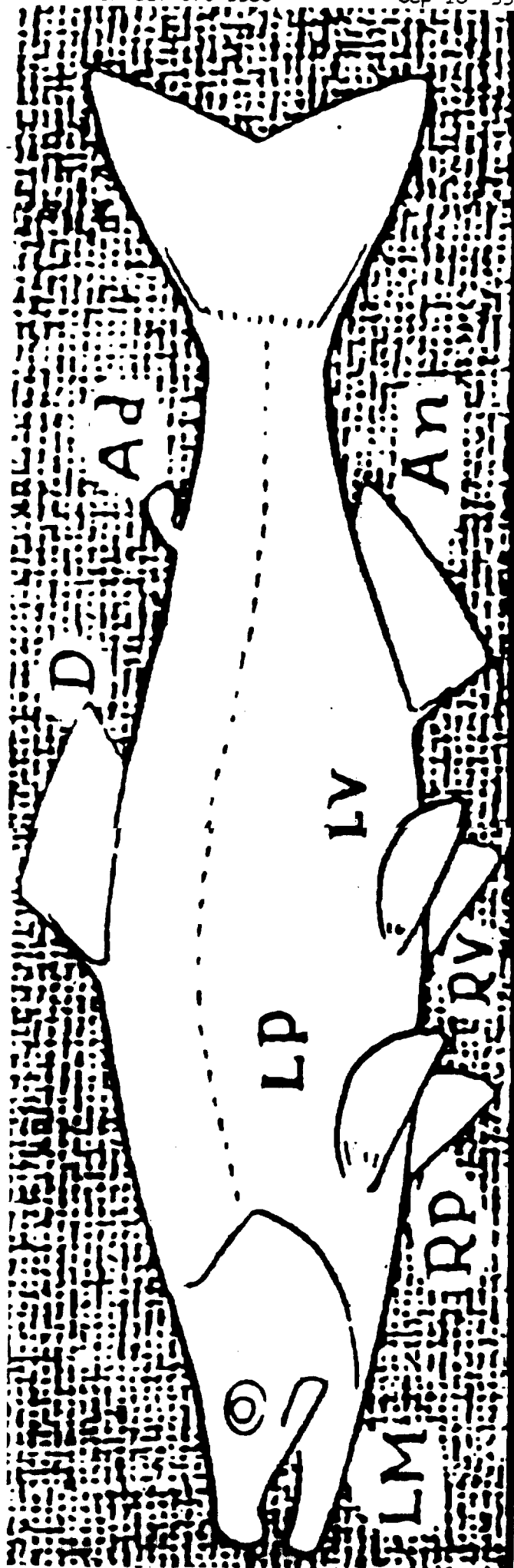
Site ID: _____ Waterbody: _____ Location: _____ Caged: _____

Collection Date: _____ **Waterbody Type:** _____ **Storet Station:** _____ **Grid:** _____

Collected by: _____ Processed by: _____ Lab: _____

[illegible]

FIN CLIP CODES



PLEASE RECORD THESE CODES ON THE SCALE SAMPLE ENVELOPES FOR TREND FISH:

D = dorsal

An= anal

RP= right pectoral

LP= left pectoral

LV= left ventral

RV= right ventral

Ad= adipose

IF MORE THAN ONE FIN IS CLIPPED, RECORD THEM ALL (ie. LPAd = left pectoral/adipose)

Attachment 8

Procedure for Collecting Samples for Aging Fish

1. Scale samples should be taken from the appropriate location on the fish (see diagram below). Take the knife point and pull the scales away from the skin. Do not take scales by scraping against them.
2. Scale samples are not adequate for aging some fish species. For the species listed below collect the appropriate items as indicated.

Walleye - scale sample and the dorsal fin spines

Carp - scale sample and dorsal fin spines

Lake Trout - otoliths

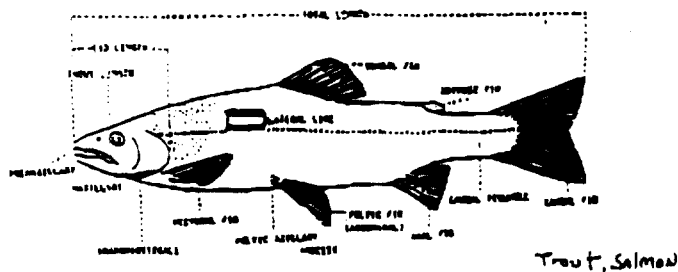
Redhorse Sucker - pectoral fin spines

Sturgeon - pectoral fin spines

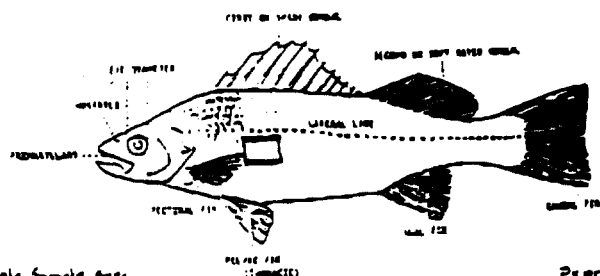
When collecting the dorsal and pectoral fin spines de-articulate the spine from the fish, do not cut the spines. De-articulating is what you are doing when you pull a drumstick off a whole chicken. This is important because we need to get the base of the spine to get an accurate age reading.

3. Samples should be placed in scale sample envelopes and the pertinent information as indicated below should be filled out.

SKETCHES OF ANATOMICAL FEATURES



Trout, Salmon



Perch non-salmonid

Coll. No. <u>XXX</u>	Serial No. _____	Grid No. _____
STATE OF MICHIGAN DEPARTMENT OF NATURAL RESOURCES		
Species <u>XXX</u>	Pin clip/ing <u>XXX</u>	
Lake <u>XXX</u>	Leisurely marks Fresh _____	OM _____
Locality <u>XXX</u>		
Gear _____	Depth _____	
Total length <u>XXX</u>	Female weight <u>XXX</u>	
Fork length _____	Dressed weight _____	
Male <u>X</u> Female <u>X</u> Mature _____	Measure _____	Size _____
Date <u>XXX</u>	Collector _____	

8-9148

☐ Scale Sample Area

OTOLITH COLLECTION PROCESS

Remove gills and scrape away the soft tissues at the base of the brain.

Locate knife across pseudobranchs inside the gill covers, slightly posterior to the point of the arrow-shaped bony structure (prootic bone) at the base of the brain cavity where the vertebral column begins (exposed portion of prootic bone).

Apply enough pressure on the knife to sever midway through pair of bulla in the prootic bone taking care not to cut all the way through the cavities containing the two otoliths. Break the prootic bulla bone open as you would a single shot shotgun, being careful not to tear the fish in half. Each of the otoliths should now be readily observable nestled in its cavity. If the lake trout was subdued with too much zeal (clubbed too hard), the contents of the cavities may be bloody, making otoliths nearly impossible to locate. The moral of this is, use just enough force to subdue the fish.

Pick each otolith out with a pair of forceps and put them on the back of your fillet gloved hand. Tease all the soft tissues of the sacculus away from the otoliths until they are stripped clean of any membranes. Now pick them off the glove and place them in a scale sample envelope for storage.

*Richard J. Jenson, Fisheries Boat Captain
Michigan Dept. of Natural Resources
3/93*

Attachment 9

Standard edible portions of Michigan's sport and commercial fishes.

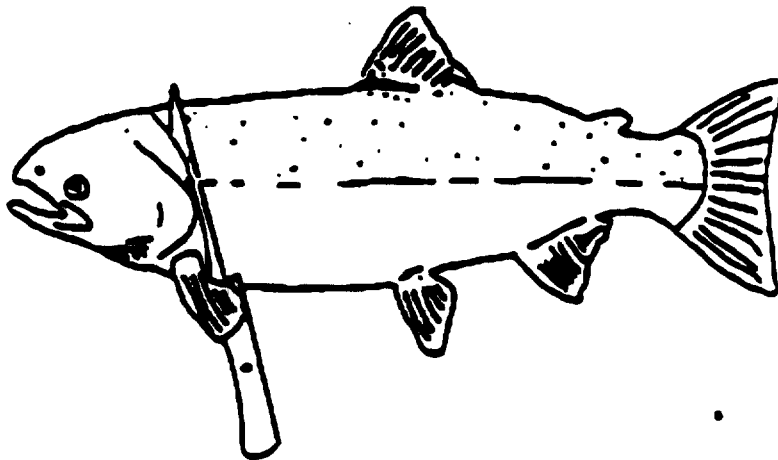
Listed below are the "standard edible portions" for Michigan fishes. The "standard edible portion" will be used for preparing fish for contaminant analyses. The "standard edible portion" is that portion of the listed species of fish that people generally eat.

Standard Edible Portion	Common Name	Scientific Name
Skin-on	Yellow Perch	<u>Perca flavescens</u>
	Walleye	<u>Stizostedion vitreum</u>
Fillet	Sauger	<u>Stizostedion canadense</u>
	Largemouth Bass	<u>Micropterus salmonide</u>
	Smallmouth Bass	<u>Micropterus dolomieu</u>
	Bluegill	<u>Lepomis macrochirus</u>
	Pumpkinseed	<u>Lepomis gibbosus</u>
	Rock Bass	<u>Ambloplites rupestris</u>
	White Bass	<u>Morone chrysops</u>
	Black Crappie	<u>Pomoxis nigromaculatus</u>
	White Crappie	<u>Pomoxis annularis</u>
	Green Sunfish	<u>Lepomis cyanellus</u>
	Longear Sunfish	<u>Lepomis megalotis</u>
	Warmouth	<u>Lepomis gulosus</u>
	Sucker Family	<u>Catostomidae</u>
	Lake Whitefish	<u>Coregonus clupeaformis</u>
	Lake Trout (lean & siscowet)	<u>Salvelinus namaycush</u>
	Rainbow Trout (Steelhead)	<u>Oncorhynchus mykiss</u>
	Brown Trout	<u>Salmo trutta</u>
	Brook Trout	<u>Salvelinus fontinalis</u>
	Splake	<u>Salvelinus fontinalis</u> X <u>Salvelinus namaycush</u>
	Atlantic Salmon	<u>Salmo salar</u>
	Coho Salmon	<u>Oncorhynchus kisutch</u>
	Chinook Salmon	<u>Oncorhynchus tshawytscha</u>
	Pink Salmon	<u>Oncorhynchus gorbuscha</u>
Skin-off	Black Bullhead	<u>Ictalurus melas</u>
	Brown Bullhead	<u>Ictalurus nebulosus</u>
Fillet	Yellow Bullhead	<u>Ictalurus natalis</u>
	Channel Catfish	<u>Ictalurus punctatus</u>
	Muskellunge	<u>Esox masquinongy</u>
	Northern Pike	<u>Esox lucius</u>
	Round Whitefish (Menominee)	<u>Prosopium cylindraceum</u>
	Lake Herring	<u>Coregonus artedii</u>
	Chubs	<u>Coregonus hoyi</u>
	Carp	<u>Cyprinus carpio</u>
	Freshwater Drum (Sheepshead)	<u>Aplodinotus grunniens</u>
	Buffalo	<u>Ictiobus cyprinellus</u>
	Burbot	<u>Lota lota</u>
	Quillback	<u>Carpiodes cyprinus</u>
Skin-off Steak *	Sturgeon	<u>Acipenser fulvescens</u>
Headless, Guttled	Rainbow Smelt	<u>Osmerus mordax</u>

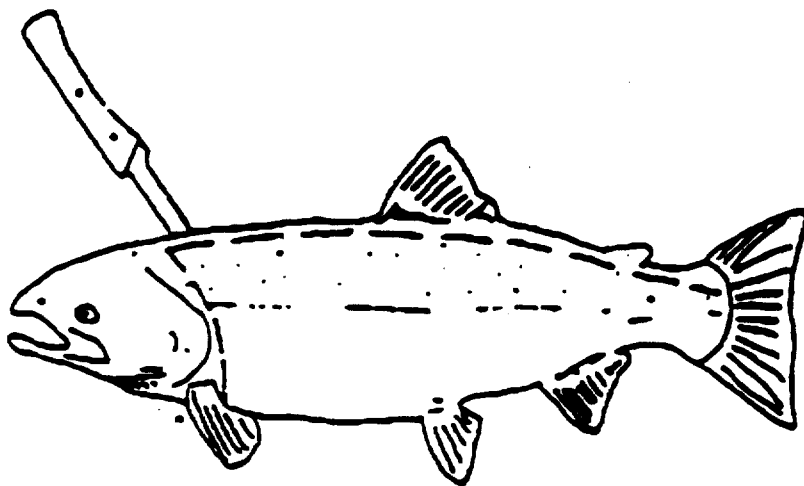
* 3" wide full cross section from the area 9-12" anterior to the dorsal fin

Attachment 10

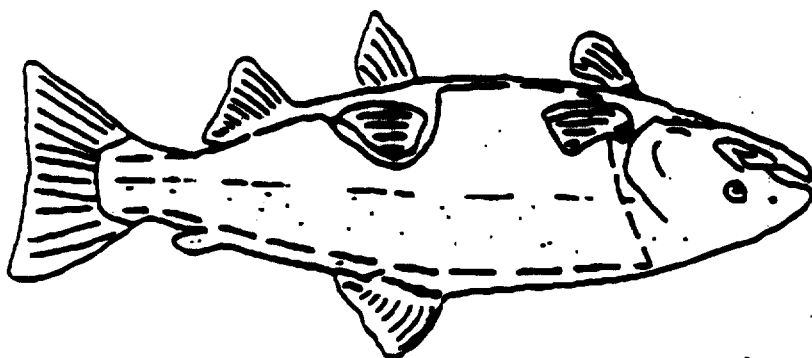
1. Make a cut behind the entire length of the operculum (gill cover) cutting through the skin and flesh to the spinal column. Dorsal to ventral cut.



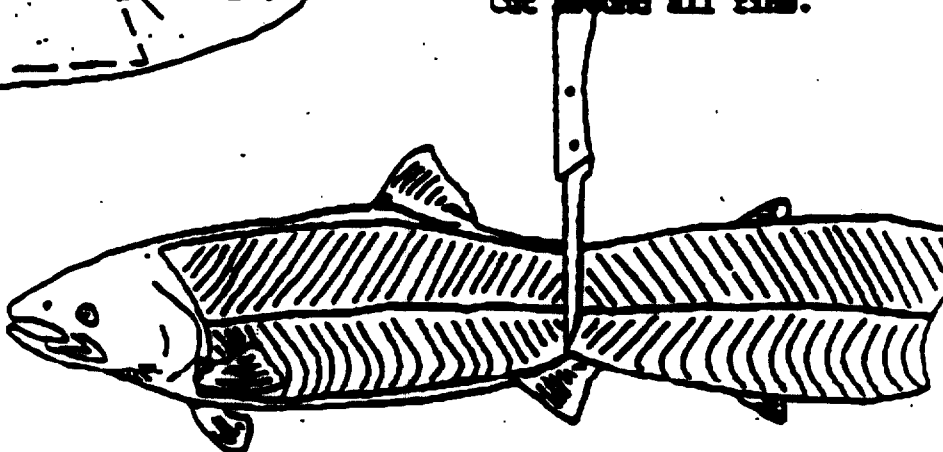
2. Make a shallow cut through the skin (to spinal column) from the base of head to the posterior end of the caudal peduncle.



3. Make a ventral cut along the belly from the base of the pectoral fin to the posterior end of the caudal peduncle. Cut around all fins.



4. Remove the fillet and then remove any major bones.



Attachment 11

MICHIGAN DEPARTMENT OF NATURAL RESOURCES
MICHIGAN DEPARTMENT OF PUBLIC HEALTH

FISH CONTAMINANT MONITORING PROGRAM
LABORATORY ANALYSIS REQUEST FORM

Water Body: _____ Site ID: _____

Location: _____

Contact Person: _____ Send Results to: _____
Address: _____ Address: _____

SAMPLE NUMBER	FIELD I.D. NUMBER	SPECIES	W/F
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			

Analysis

Lipids Organic & Mercury Mercury Dioxins & Furans

Special Analysis: _____

Appendix C
**SOPs for Collecting Bedded Sediment,
Bedload Sediment, and Settling Sediment**

STANDARD OPERATING PROCEDURE

COLLECTING SEDIMENT SAMPLES USING A HAND-HELD VIBROCORING UNIT

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes a technique for collecting short (< 6') sediment cores using a battery-operated, hand-held vibrocoring unit. The cores are 2" in diameter, and collected in tubes made of either cellulose acetate butyrate (CAB) or Lexan plastic. Sediment cores are useful for investigating the vertical distribution of contaminant concentrations, for evaluating contamination histories, assessing the volume of contaminated sediment, etc. Much of the information in this SOP is contained in Smith, et al. (1996).

2.0 METHOD SUMMARY

A sediment core is collected using a battery-operated, hand-held vibrocoring unit, in 2"-diameter core tubes. The core tube is opened using a saw, and sediment subsamples collected as per the project-specific Field Sampling Plan. This vibrocoring unit is not submersible, and is best used in waters less than 10' deep.

3.0 SAMPLE COLLECTION AND PROCESSING

3.1 Supplies and Materials

- a hand-held vibrocoring system, designed by AScI Corporation, consisting of: electric vibrator motor (12 V DC) and mounting plate with socket for attachment of 2" diameter extension poles
- two, 12 V DC storage batteries with charger
- core tube adapter and clamp with check valve and retrieval lines attached
- 2-10 ft. extension poles
- 6.5 ft. (2 meter) lengths of 2" diameter core tube (CAB [cellulose acetate butyrate] polymer) with or without CAB core catchers attached
- 2" diameter PE (polyethylene) end caps
- duct tape
- waterproof marker pens
- portable drill and 3/8" bit
- tube cutter tool
- glass or polypropylene sample bottles
- field crew of at least 2
- this SOP

3.2 Collecting the Core

- 1) Locate the sampling station with an appropriate field positioning system that provides suitable accuracy (~ 3 to 5 m).
- 2) Measure the water depth using appropriate means, such as a sounding line, marked pole or fathometer.
- 3) Check for secure attachment of the retrieval lines to the core tube mounting clamp.
- 4) Insert a 6.5 ft. length of 2" diameter CAB core tube (core catcher end down) into the mounting clamp and tighten the four wing nuts securely by hand. Make sure clamp is tightened evenly.
- 5) Choose an extension pole of appropriate length (water depth or longer) and insert it into the mounting plate socket; secure it using a 1/4" bolt and locknut.
- 6) Slip the flared lower end of the extension tube over the check-valve end of the core tube adapter, and hold it on by applying upward tension on the retrieval lines. Lower the system vertically (CAB tubing first) into the water to the bottom. Press and vibrate tube into the sediment until it is inserted 6 ft., or until refusal occurs. Note insertion length by markings on extension pole.
- 7) Disengage the extension pole and stow on board sampling vessel.
- 8) Retrieve the core tube containing the sample by pulling on the two retrieval lines, either manually or by using a davit-mounted hand winch.
- 9) Slip the core tube cap over the lower end of the core tube, and secure with duct tape.
- 10) With tube and barrel held vertically in the boat, drill hole in tube just above the top of the sediment column to drain off water.
- 11) Cut off the tube just above the sediment surface and cap the upper end.
- 12) Label the tube lengths with sample station ID codes with a permanent marker. Also, make sure the upper end is marked as such.
- 13) Transport core ashore for processing as soon as possible. Core may be stored within a cooler or enclosed box with bag ice, if it is short enough. This is rarely necessary, unless volatile organics are contaminants of interest.

3.3 Processing the Core

The sediment core is usually processed in a nearby field facility in order to describe its structure and create subsamples for chemical analysis. This is important to document the core content and to maintain sample quality.

- 1) Cut the CAB core liner (filled with sediment) lengthwise along opposite sides.

(Note: cut through the liner wall without cutting significantly into the sediment core itself. Disturbed sediment adjacent to the liner wall should not be sampled anyway, but it is important not to contaminate the undisturbed interior of the core with plastic chips or other debris from the cutting process. If, before coring, the outer wall of the CAB liner (1/16" thick) is scored or pre-cut halfway through with a circular saw or other tool, then the final cut during processing can be made with a razor knife. However, CAB plastic is very tough, and cutting with a razor knife can be dangerous and difficult to control without cutting into the core. The best hand tool available for cutting hard plastic liners is an electrical vibrating or "reciprocating" saw of the type used in industry to cut sheet metal, or in medical practice to cut off plaster casts. The cuttings tend to form ribbons rather than chips, which helps in avoiding contamination of the sediment inside. Also, the vibrating blade is much safer to use than a conventional saw blade, since it does not readily cut soft material such as skin.)

- 2) Once the liner wall is cut through along opposite sides (top and bottom of the horizontal core), use a spatula or a flat, thin blade of rectangular shape to cut the sediment core lengthwise into two half-cylinders. Vertical cutting in discrete steps, rather than "dragging" the blade through the core insures that the layered structure of the core is not obscured, and that contaminants are not spread across layers. Between each vertical cut, wash and scrub all adhering sediment off of the blade in a bucket of clean tap water.

(Note: it is usually not practical to decontaminate the blade fully after each cut, but any chance of contaminant carryover between zones can be minimized by cutting through the less oily parts of the core first. It helps if the blade is wet when cutting through oily silt or stiff clay sediments, which tend to adhere. A cleanly cut surface is best for documenting core structure.)

- 3) Arrange the two half-cylinders of the core section side-by-side, with the cut surfaces facing up. Extend a tape measure along beside them, starting at the original top end of the core.

- 4) (Optional) Photograph the core in color with a track-mounted 35 mm camera. With 160 watts (4, 4' bulbs) of fluorescent light, 200 speed film is suitable for good results. Insure that the wet surface of the core does not reflect light directly into the camera lens. A polarizing filter helps to reduce reflectance off the wet core surface. Photograph the core section in overlapping frames; place a small label with core field ID number so that it appears in each frame. Advance the tape measure appropriately for any additional sections of the same core.

- 5) While the core section is still intact, record a general description of the core structure, noting zones of different color, texture, sediment type (silt, sand, clay, gravel, etc.), and apparent oiliness. Intentionally smelling the core to note its odor is not recommended.
- 6) Collect each core interval, as pre-determined in the study plan, from the undisturbed core interior with a clean, stainless steel spoon or spatula. Place the sediment from an individual core interval into a clean stainless steel mixing bowl of appropriate size.
- 7) Mix the sediment with a clean stainless steel spoon until visually homogeneous. During this operation, remove any obviously "non-sediment" objects from the sample; bottle caps, broken glass, sticks, large rocks, etc.
- 8) Place an appropriate volume of sediment into a wide-mouth glass jar (pre-cleaned according to EPA protocols), leaving space at the top of the bottle for later mixing (unless the samples are for volatile organics analysis, in which case the jar should be completely filled).
- 9) Label each jar with a unique sample identification number. Store the sample bottles on ice or in a refrigerator until transfer shipment to the analytical laboratories.

4.0 HEALTH AND SAFETY AND ENVIRONMENTAL COMPLIANCE

1) Field crew personnel should always wear appropriate personal protection clothing and equipment, which at a minimum includes:

- Safety glasses or face shields
- Tyvek or Saranex coveralls
- Double gloves (Latex inner gloves and Nitrile outer gloves)
- Steel-toed rubber boots
- Life jacket, under coverall

The intention of this clothing is to minimize personal exposure to the possibly hazardous sediments.

2) Field crew personnel should make a point of consuming liquids (cool water recommended) on a regular basis to minimize heat stress during warm weather, or warm themselves regularly to avoid hypothermia during cold weather, and take periodic rest breaks to minimize fatigue.

2) Excess sample should be disposed of in an appropriate fashion, which should be described in the project Field Sampling Plan.

5.0 REFERENCE CITED

Smith, V.E., J.E. Rathbun, S.G. Rood, and L.L. Huellmantel. 1996. Technical Considerations in Sediment Quality Surveys. *J. Great Lakes Research* 22(3):512-522.

STANDARD OPERATING PROCEDURE

BEDLOAD SEDIMENT SAMPLING FOR ORGANIC CONTAMINANT ANALYSIS

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes a method for collecting bedload sediment samples. Bedload sediment is that component of the fluvial sediment load that moves in a rolling or saltating mode. The sediment particles move at a speed that is less than the velocity of the transporting flow and are confined to a layer, a few grain diameters thick, immediately above the stream bed (Gomez, 1991). This SOP emphasizes procedures for collecting bedload samples during precipitation events.

2.0 METHOD SUMMARY

A bedload sampling device is positioned on the stream bottom before a precipitation event, and retrieved afterwards. Collected sediment is removed from the nylon mesh sample bag, transferred to a clean sample jar, and submitted for analysis.

3.0 PROCEDURE

3.1 Equipment and Supplies

- bedload sampler of appropriate weight
 - current < 5'/second = 65 lb. sampler; > 5'/second = 105 lb. sampler
 - stream wadeable = hand-held 4 lb. sampler
- clean (solvent-rinsed) nylon mesh sampling bag; 125 µm or 250 µm mesh recommended
- certified clean wide-mouth glass jars; 100 mL or larger recommended
- spatulas
- appropriate supplies to securely fix sampler to stream bottom (see procedures)
- global positioning system or other means of identifying station location

3.2 Procedure

[Note: steps marked with “**” denote general descriptions of activities that will vary from project to project, the details of which are described in detail in the project-specific Field Sampling Plan.]

1. Establish position of station and record location in the field notebook.** Appropriate procedures could include use of a global positioning system, triangulation on local landmarks, careful notations on a detailed map, etc.

2. Prior to deploying the sampler, clean the mesh sample bag with appropriate solvents (see the Field Equipment Decontamination SOP for details). Rinses with methanol, n-hexane, and distilled water are recommended.
3. A day or so prior to the rain event (or at any time, if not sampling a precipitation event), securely fix the sampler to the stream bottom. Recommended procedures include:
 - hanging the sampler from a bridge, using a steel cable
 - driving fence posts or concrete reinforcement bar (re-bar) into the bottom
 - fixing the sampler to a concrete (or other) weight
 - or just relying on the weight of the sampler to hold it in place
4. Leave the sampler in place through the rain event (i.e., until the water level returns to a normal height).
5. Retrieve the sampler and remove the mesh sample bag.
6. Remove any extraneous, non-sediment material from the bag; trash, sticks, leaves, etc.
7. Transfer the trapped sediment into a labeled wide-mouth glass jar, using a spatula or other appropriate instrument.
8. Keep the sample jar on ice until shipment to the analytical lab.

4.0 FIELD QA/QC

1. If specified in the project-specific Field Sampling Plan, duplicate samples may be collected by deploying two samplers side-by-side.
2. Field rinsate blanks of the mesh sampling bag are recommended. Rinse the bag with methanol and n-hexane, into a sample jar, and analyze for the contaminants of interest.

5.0 HEALTH AND SAFETY

Normal water safety precautions must always be observed, including wearing life vests, taking care to avoid hypothermia or heat stroke, etc.

6.0 REFERENCES

Gomez, B. 1991. Bedload Transport. *Earth Science Reviews* 31:89-132.

STANDARD OPERATING PROCEDURE

COLLECTING SETTLING SEDIMENT USING A SEDIMENT TRAP

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes a method for collecting settling sediment – sediment (or similar materials like plankton) temporarily suspended in the water column, but which is slowly settling towards the bottom (bedded) sediments. Settling sediment is usually collected in lentic environments (ponds, lakes, or impoundments), not lotic environments (running streams and rivers). Temporarily suspended sediment in lotic environments is termed bedload sediment.

This SOP can be used to collect settling sediment samples associated with short-term precipitation events or longer-term monitoring.

Note: parts of this SOP are derived from Blomqvist and Kofoed (1981); Cripps and Clarke (1998); Eadie, et al. (1984); Dobson and Mackie (1998); and Larsson et al. (1998).

2.0 METHOD SUMMARY

Sediment traps (described in Section 3) are suspended from buoys and left in a water body until they have collected a sufficient mass of settling sediment. This mass will vary depending on the analyses performed; two grams is usually a minimum.

3.0 PROCEDURE

3.1 Materials and Supplies

Note: Sediment trap efficiency is dependant on the trap's aspect ratio (height:diameter) and the absolute diameter of the opening. The most efficient collectors are relatively tall, narrow cylinders with a height:diameter ratio of ≥ 3 (Blomqvist and Kofoed, 1981).

- Sediment traps; 1-quart (or 1,000-mL) pre-cleaned flint glass jars
- An appropriate harness to hold the traps to the buoy line
- A buoy of sufficient buoyancy to suspend the traps at a consistent water depth
- An anchor of sufficient weight to prevent the buoy and traps from drifting off station
- An appropriate sampling vessel (project-specific)
- Appropriate equipment for establishing station location; a global positioning system (GPS), etc. (project-specific)
- Ice chests

3.2 Procedure

1. Anchor boat on the sampling station, and establish position using GPS or some other means.
2. Secure the sediment trap jars to the harness (= the trap assembly), and secure the harness to the buoy rope. Three or more traps are recommended, to obtain adequate material for analysis (Dobson and Mackie, 1998).
3. Lower trap assembly to an appropriate depth, and secure to buoy. It is recommended that the traps be positioned in the water column closer to the water surface than to the bottom sediment, to minimize collection of resuspended bottom sediments rather than settling sediment.
4. Leave the trap/buoy assembly on site for an appropriate period. A period of up to one month is recommended for sampling settling sediment under dry weather conditions, while a period of only 1 to 3 days may be adequate for storm event-related sampling.
5. Upon retrieval, pour off overlying water from each trap, leaving the settled sediment in the bottom of the jar.
6. Label and cap jar, put jar into a ziplock plastic bag, and place in an ice chest. Lay bottles on their sides to prevent breakage if contents freeze.
7. Keep jars on ice until transferred to the analytical laboratory.

4.0 FIELD QA/QC

Details of the field QA/QC procedures will be project-specific and should be described in the project Field Sampling Plan. Typical field QA/QC operations include collection of duplicate samples at regular intervals (a certain number per day or per water body sampled, or 1 per 10 or 20 samples).

5.0 HEALTH AND SAFETY

Normal water safety precautions must always be observed, including wearing life vests, taking care to avoid hypothermia or heatstroke, etc.

6.0 REFERENCES

Blomqvist, S., and C. Kofoed. 1981. Sediment Trapping – A Subaquatic *In Situ* Experiment. *Limnol. Oceanogr.* 26:585-590.

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Appendix D

Bioaccumulation Studies Using Caged Fish

STANDARD OPERATING PROCEDURE

IN SITU BIOACCUMULATION STUDIES USING CAGED FISH

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes conducting an *in situ* bioaccumulation study using caged fish. This procedure is applicable to any water body; stream, lake, wetland, etc. Young-of-the-year (YOY) channel catfish (*Ictalurus punctatus*) or fathead minnows (*Primephales promelas*) are recommended.

2.0 METHOD SUMMARY

Caged YOY fish are deployed in cages for 28 days, and retrieved for contaminant analysis. Fish are processed as whole fish, not fillets.

3.0 PROCEDURE

3.1 Equipment and Supplies

- YOY fish, from a commercial hatchery or other source known to be free of contaminants
- metal mesh cages; approximately 18" x 18" x 30" recommended
- appropriate supplies to secure the cages on-station; see below
- certified clean wide-mouth glass jars; 500 mL recommended
- an appropriate boat for deploying the cages
- ice chests
- global positioning system, or other means of establishing station locations

3.2 Procedure

[Note: steps marked with "***" denote general descriptions of activities that will vary from project to project, the details of which are described in detail in the project-specific Field Sampling Plan.]

1. Establish position of station and record location in the field notebook.** Appropriate procedures could include use of a global positioning system, triangulation on local landmarks, careful notations on a detailed map, etc.
2. Measure the dissolved oxygen in the water at the depth at which the cages will be deployed. If it is less than 3 mg/L. consider moving the station.
3. Fix the cage on-station by an appropriate means. This could include:
 - attachment to a bridge piling, or other permanent structure
 - suspension from a buoy, which is attached to an anchor

4. While the cage is partly in the water, carefully transfer the fish to the cage, taking care not to injure them. Ten to twenty fish are recommended per cage, depending on the size of the cage.
5. Recommendation: Suspend the cage in the water column, so that it is not in contact with the sediment, or below any thermocline that might form during the exposure period. Warning: Take what ever steps are possible to minimize vandalism during the exposure period.
6. Leave the cage on-site for 28 days.
7. At the end of the exposure period, retrieve the cage and transfer the fish to one or more labeled wide-mouth jars.
8. Place the jars in an ice chest on ice and transfer to the analytical laboratory.

4.0 FIELD QA/QC

Depending on the intentions of the study, it may be desirable to process the fish from each cage as multiple samples rather than a single sample. This assumes that sufficient fish survive the exposure. If sufficient fish are available, triplicate samples are recommended for investigating contaminant heterogeneity. Information on sample replication requirements are stated in the project-specific Field Sampling Plan.

5.0 HEALTH AND SAFETY

Normal water safety precautions must always be observed, including wearing life vests, taking care to avoid hypothermia or heat stroke, etc.

Appendix E
Deployment of Semipermeable Membrane
Devices

STANDARD OPERATING PROCEDURE

DEPLOYMENT OF SEMIPERMEABLE MEMBRANE DEVICES

1.0 SCOPE AND APPLICATION

This procedure describes the procedures for deploying and collecting caged semipermeable membrane devices (SPMDs), for assessing bioavailable concentrations of hydrophobic, lipophilic organic contaminants (e.g., PCBs, pesticides, etc.). SPMDs accumulate dissolved-fraction organic contaminants, which are the contaminant fraction most available to aquatic organisms. Procedures are described for both commonly used types of SPMDs; those filled with triolein and those filled with n-hexane (e.g., the PISCES sampler).

As with any field deployment, care must be taken to balance the need for a secure, vandal-resistant deployment vs. the need to retrieve the samplers.

2.0 METHOD SUMMARY

SPMDs are deployed in metal cages in a water body for 28 days, retrieved, and processed for organic contaminant analysis.

3.0 PROCEDURE

3.1 Materials and Supplies

- SPMDs; either the type filled with the triglyceride lipid triolein, or the solvent n-hexane.
- Appropriate metal cages, preferably made of stainless steel.
- Appropriate deployment supplies; buoys, ropes, anchors, hose clamps, etc. The exact supplies required will vary from one location to another.
- Paper towels
- Ice chests
- Wide-mouth jars or metal cans, for transporting the SPMDs

3.2 Procedure

[Note: steps marked with "***" denote general descriptions of activities that will vary from project to project, the details of which are described in detail in the project-specific Field Sampling Plan.]

1. Transport the SPMDs to the field, taking care to minimize exposure to the atmosphere or other potential sources of contamination. Triolein-filled SPMDs may be transported in their metal shipping cans.

2. Establish position on station and record location in the field notebook.** Appropriate procedures could include use of a global positioning system, triangulation on local landmarks, careful notations on a detailed map, etc.
3. Expose one or more SPMDs to the atmosphere for the duration of the deployment process. This sampler will serve as an atmospheric blank, and should be processed like the exposed samplers.
4. Transfer the SPMDs from their transportation containers to pre-cleaned cages, as necessary. (This is usually necessary for triolein-filled SPMDs, and unnecessary for hexane-filled PISCES samplers. ~~Create field replicate samples as required by the~~ Field Sampling Plan.**)
5. Lower the caged SPMD or PISCES samplers into the water to an appropriate depth, and secure in place.** Appropriate options include:
 - Suspension from a buoy
 - Attachment to a concrete block that is lowered to the bottom
 - Attachment to a permanent structure like a bridge piling
6. After deployment is complete, mark the station so that it may be found at the end of the sampling period.** Stakes, flags, buoys, etc., may be used. Consider securing the deployment device to the shore or a permanent structure such as a bridge piling, if appropriate.
7. Leave the samplers in place for 28 days, or another time period if required by the study.
8. (Optional) During the deployment it may be desirable to temporarily retrieve the samplers for inspection. This is especially advisable for triolein-filled SPMDs, which are prone to biofouling by bacteria and algae, and should be cleaned after 14 days using a paper towel.
9. At the end of the exposure retrieve the samplers, remove them from their cages (if necessary), clean the samplers with a paper towel (if necessary), and place the samplers in a clean glass jar or metal can. Label the container with station number, date, etc.
10. Place the containers on ice, and transfer to the analytical laboratory.

4.0 FIELD QA/QC PROCEDURES

1. Replicate SPMDs are usually deployed at each station, to assess contaminant heterogeneity. Triplicate SPMDs are often used.

2. SPMDs should be protected from unnecessary exposure to the atmosphere, since they can accumulate measurable amounts of contaminants from the air. An "air blank" should be collected in the course of any field deployment, as described in Section 3, Step 3.
3. Biofouling can be a significant problem with SPMDs. Triolein-filled SPMDs are especially susceptible, and should be inspected once or twice during the 28-day exposure period and cleaned by wiping with a paper towel as necessary. The PISCES sampler, filled with n-hexane, is less susceptible to biofouling because the hexane permeates the membrane and inhibits biological growths.
4. Diffusive contaminant uptake in aquatic systems is proportional to the passage of water across the membrane surface. When deploying SPMDs at multiple locations, it is important to place them in locations with similar flow regimes.
5. Diffusive uptake is also directly proportional to water temperature, so it is advantageous to deploy the SPMDs at a time of year when the water temperatures are high and more or less constant.

5.0 HEALTH AND SAFETY

Each field deployment, and the associated hazards, are unique. The project-specific Health and Safety Plan should explain all expected hazards and procedures to cope with them. Water safety issues are the most common hazards encountered in SPMD deployments, and normal water safety precautions must always be observed, including wearing life vests, taking care to avoid hypothermia or heat stroke, etc.

Exposure to the hexane in the PISCES samplers should be a minimal hazard, because the volume is so small (approximately 200 mL).

6.0 SELECTED REFERENCES

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Appendix F

Field Equipment Decontamination

STANDARD OPERATING PROCEDURE

FIELD EQUIPMENT DECONTAMINATION

1.0 SCOPE AND APPLICATION

This procedure is applicable to removing organic contaminants from reusable field equipment used to collect water, fish, sediment, or soil sediments.

2.0 METHOD SUMMARY

Equipment is sequentially washed with a detergent and then rinsed with polar and nonpolar solvents, and water. Clean equipment not immediately reused is wrapped in solvent-rinsed aluminum foil, or otherwise protected from recontamination.

3.0 PROCEDURE

Note: much of the following information is taken from U.S. EPA/U.S. ACOE (1995).

3.1 Materials and Supplies

- Distilled water
- Non-phosphate soap (e.g., Alconox)
- Reagent-grade methanol
- Reagent-grade n-hexane
- "Like-rags" or paper towels
- Aluminum foil
- Scrub brushes
- Garbage bags
- Zip-lock bags
- Basins to wash in and collect rinsates

3.2 Procedure

[Note: This cleaning procedure should be applied to appropriate equipment at a frequency (between stations; between between sampling transects; etc.) specified in the project-specific field sampling plan.]

Preparation

Prepare the following waste containers: waste baskets lined with plastic garbage bags for paper towels; 3 basins for soapy water, tap water rinses, and solvent rinses.

Cleaning Procedure

- 1) Wipe off all visible materials using "Like-rags" or paper towels.

- 2) Scrub with Alconox and tap water.
- 3) Rinse once with distilled water.
- 4) Rinse once with methanol.
- 5) Rinse once with n-hexane.
- 6) Rinse 3 times with distilled water.
- 7) Allow to air dry and wrap small equipment in aluminum foil.
- 8) For longer term storage, small equipment (spatulas, knives, spoons, bowls, etc.) can be stored in zip-lock bags. Larger equipment (Ponar grabs, water samplers, etc.) can be wrapped in larger plastic bags like garbage bags, or stored closed in their field carrying cases.

4.0 CITED REFERENCE

U.S. EPA and U.S. Army Corps of Engineers. 1995. *QA/QC Guidance for Sampling and Analysis of Sediments, Water, and Tissues for Dredged Material Evaluations*. EPA 823-B-95-001. U.S. EPA Office of Water, Washington, DC. 131 pp. + appendices.